COMMISSION OF THE EUROPEAN COMMUNITIES

B. C. R.

STANDARDS AND REFERENCE SUBSTANCES

(Direct action)

BALANCE ACCOUNT FOR 1974

1975 EUR 5403 e

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by

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Joint Nuclear Research Centre
Ispra Establishment - Italy

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ABSTRACT

This document describes in an abridged fashion the work which was performed in the JRC-Ispra laboratories for the objective "Standards and reference materials" during the year 1974. Besides a technical support to the BCR secretariat in Brussels concerning the indirect action, an important laboratory activity took place in several fields:

— taking part in the setting up of various reference methods and expertising for the various services of the Commission;

— analysis of several compounds for certifying their composition and/or their purity (electrolytical Cu, O_2 and N_2 in metals, precious metals in Cu, coke, atmospheric dust, trace elements in animal, vegetable and coal matrices;

— setting up the equipment and first trials for the determination of viscosity and thermal conductivity of solids and liquids;

— continuation of the work in the technological field (friction, fault detection by ultrasonics, toughness).

This report has besides an annex concerning the state of development of the data bank on reference materials and their users.

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Ispra, February 12th, 1975
HL/cf-00800/1975

STANDARDS AND REFERENCE SUBSTANCES

Progress Report for
1974

In 1974 our laboratories developed and extended the activities officially undertaken during 1973 [1], and at the same time assembled equipment more specifically suited to the needs of a programme which is highly demanding from the point of view of precision and accuracy. While continuing their experimental work, various specialists of the Ispra Establishment, occasionally supplemented by specialists from the Geel Establishment (Belgium), made a significant contribution to the definition of a broad European programme to be carried out in national laboratories (indirect action) and Commission laboratories (direct action).

Furthermore, those responsible for the "Standards and Reference Substances" objective put a great deal of work into the definition of new activities for the JRC Petten Establishment (Netherlands). This work has based on:

1) The conclusions drawn from the general enquiry on needs for reference materials [2]

2) The recommendations made during the Round Table discussions on "Reference materials for organic chemistry [3]

3) Preliminary orientations proposed by the Working Sub-groups "Analyses of organic products" [4], "Plastics and rubbers" [5] and "Petrol and allied products" [6]

4) The need of various Services of the Commission for technical expertise in the field of petroleum products, foodstuffs, cosmetics, etc.

The JRC has proposed to entrust Petten with activity of the "Standards and Reference Substances" type, but more specifically devoted to chemical analyses and measurements of properties of organic materials. The corresponding documents were subsequently submitted to the General Advisory Committee [7] and to the Advisory Committee concerned with the Management of the BCR Programme [8]. Following the recommendations made by these Committees, the Commission transmitted a formal proposition to the Council [9] which, if it is accepted, will lead to the

direct action being reinforced by a staff of the order of 15 men/year, not including infrastructure.

While awaiting the appearance of more detailed reports, the present document briefly described the results obtained during 1974, specifying the role played by several specialists of the JRC, in defining the "indirect actions" to be executed in public and private national laboratories. Moreover, with each activity, one can see in how far our laboratory work has been technically integrated into a network of far greater range.

We have divided into five actions, each of which includes a considerable number of subjects, the work of Ispra personnel engaged fully or partially in this objective:

-Support to the BCR secretariat
-Support to the Services of the Commission
-Reference materials and chemical analysis
-Reference materials and physical properties
-Reference materials and technological properties

REFERENCES

1) Communication(in printing); see also Preprint of 7/2/74 HL/cf-00800/3091/3

2) Report EUR 4886 d,f,i,n,e,(1972)

3) Proceeding of the meeting of 20/2/74, doc. BCR/39/74 of 19.3.74

4) Draft minutes of the meeting of 25/9/74, doc. BCR/102/74 of 3/10/74

5) Draft minutes of the meeting of 3/10/74, doc. BCR/103/74 of 9/12/74

6) Draft minutes of the meeting of 14/11/74, doc. BCR/6/75 of 10/1/75

7) CCG 137 rev. 1 plus appendixes

8) EUR/C-IS/585/74,e,f of 7/10/74

9) COM (74) 2200 final plus the appendixes of 18/12/74.

1. SUPPORT TO THE BCR SECRETARIAT

As has already been mentioned in a previous report [1], various staff members and specialists of the JRC are devoting all or some of their time to the activities which reach far beyond the boundaries of their Establishment.
Thus it is that, according to their individual competences, these specialists take care of secretariats and/or lead Working Group and Sub-Groups, whose compositions and scopes have been only slightly modified since last year.
On the other hand, new groups of national experts were set up during 1974 within the framework of indirect action, and the JRC, lacking available qualified personnel, is very poorly represented in these.

In addition to the above, various activities of general interest have been going on during the year:

-Guide Book for addresses for users of reference materials
-Data Bank on reference materials and organisations involved in thse products
-Information Bulletin
-Applied statistics

1.1. Guide Book for addresses (Maurandy C.)

Following limited distribution of a provisional edition, our services incorporated improvements and distributed something like 2.000 copies of the first edition of this document [10].
According to the reactions already received, this guide book seems, on the whole, to have been well received, and we are already planning for the end of 1975 or early 1976, the compilation of and improved and updated second edition. Needless to say, work on gathering the necessary materials has already started.

1.2. Data Bank (Maurandy C., Bertolo C.)

With the idea of one day setting up an Information Bureau in the framework of the BCR, the Ispra Services have undertaken to compile a computerised data bank, which could eventually be linked up with a European teleinformation network (action COST II). This bank has been the subject of an internal report [11] of which a slightly revised version can be found in Appendix I. Undertaken in 1973, the compilation of this thesaurus (key-words and bank structure) has been continued into the first half of 1974. Subsequently, and relying on the questionnaires of the general enquiry and the pharmaceutical enquiry, 1330 objects of the class AAAD (addresses of suppliers and users of reference materials) have been memorised (punch cards, and magnetic tapes).
As from the month of June, and basing our work on the technical documentation in our possession, we have prepared and fed in 4.330 objects of

the class AAQY. This class includes, in a codified form, the technical characteristics of reference materials as well as the names of their manufacturers and distributers.

In addition, 180 objects of the class AA8K, have been recorded; they involve the names, addresses, functions and other characteristics of national experts, participating in the development of the BCR action (members of the ACPM, the Working Groups and Sub - Groups, etc..)

During 1975 the memorisation of the data already in our possession, will continue with updating whenever necessary, and the improvement of certain details of the programme which should facilitate exploitation of the bank, and make the presentation of the results clearer. Also envisaged for 1975 is the setting up of direct liaison between a terminal at Brussels in the BCR secretariat, and the Ispra computer. It will thus be possible to study, both in the hardware and software sectors, the possibility of a remote dialogue between the bank and the user.

1.3. Information Bülletin (Mosselmans Nicole)

After examining the second mock-up[12] presented by the Ispra Services in June 1974, the ACPM recommended, during its second meeting [13] that the BCR should publish, as early as possible, a simplified version of this bulletin, including in particular:

- information on the various activities being carried out within the framework of the BCR (direct and indirect action)
- information on the manifestations involving activities linked to those of the BCR
- bibliographic lists, if possible detachable, including the references and summaries of publications dealing with the preparation, certification, and utilisation of reference materials
- an annual index of the bibliographic lists which have already appeared in the bulletin.

The first issue will include, amongst other things, a series of articles describing the aims, organisation and work in progress or in preparation in the framework of the BCR. Only in a second stage will there be any plans to supplement this bulletin with scientific or technical articles concerning, for the moment, reference materials and methods. By early 1975, it is hoped to form an Editing Committee, composed of representatives of the General Directions concerned, who will supervise the operation as a whole.

While awaiting the setting-up of this structure, the Ispra Services have already studied several mock-ups of this bulletin, defined a diagramme of the operations to be carried out, and estimated the necessary resources (see Annexes II and II bis) and selected roughly 600 references and summaries of publications, taken from the compilation of about 55 reviews and periodicals and from 24 "abstracts" and descriptive bulletins (between 2.000 and 3.000 reviews). The compiled list of

these documents is given in Annex III.

It is abvious that we are still far from covering all the literature which might be of interest to the BCR action, and that some agreement will have to be made with national organisations to enlarge our field of action. Furthermore we have established a list of already selected references, and have gone on to preliminary tests of the use of the KWIC system for the automatic establishment of these lists, and the periodical editing of the selected references. These lists will enable us, moreover, to verify whether or not the same reference has been entered twice. Without describing the KWIC system in detail, since it is so well known[14], we will only mention that the listings enable us, by means of "significant words" taken from the title and/or content of the selected documents, to identify rapidly a reference of interest to the user, and appearing in this or that number of the information bulletin. Since these listings are only in English, we have to translate into this language all the titles, summaries, and "significant words" of the documents printed in other languages.

We hope that 1975 will see,together with the continuation of document selection, an improvement in the presentation of the listings, a simplification of the utilisation of the "significant words", and the edition of a preliminary cumulative list of the selected references.

1.4. Applied Statistics (J. Larisse)

The statistical activities concern mainly applications and development of designs of experiments in order to define and to propose, if possible, the use of the best statistical methods in this field.
During the year 1974, two main activities have been developed.

1.4.1. Theoretical Studies on the actually existing methods to be handled in order to assume the best service to the users

It can be shown that the classical analysis of variances cannot be used because one of the mathematical base hypothesis, the equality of variances, is not verified in most cases. Consequently, we have developed and used an intercomparison technique based on the so-called Aspin-Welch test. There exists other possibilities which have been discussed briefly during the first meeting of the Working Group "Statistical Principles" held in Brussels the 14th of May 1974. A second meeting devoted to a more detailed discussion on the different approaches proposed is foreseen. Roughly speaking it can be concluded that the Aspin-Welch technique is as valid as the other methods. However, if some further information can be gathered from the probability distribution of the measurement errors, this information can be used to improve the statistical calculus. The problem would be to estimate the "cost" of this information.

1.4.2. Applications of the BCR technique

Until now, the Aspin-Welch technique has been discussed and used in each of the following working groups:

a) Work in connection with the Eurisotop Bureau; determination of O_2 in non-ferrous metals

-meeting of the chairmen of the working groups O/Cu, O/Pb, O/Zr, 12/2/74

-4th meeting of the working groups 0/Cu, O/Pb, O/Cu Desox, 5/3/74

-4th meeting of the working group O/Zr, 4 and 5/6/74 (Larisse)

-4th meeting of the sub-group "Charged particules and photons γ"; 5th meeting of the working group O/cu, 26 and 27/11/74.

b) Determination of "Commercial" elements in ore samples and impurities in electrolytic copper

-meeting of 21/5/74

-meeting of 1/10/74

c) Work in connection with environmental pollution problems (Girardi)

-meeting of the 7 and 8/10/74 (Larisse-Fangmeyer-Haemers)

d) Work in connection with the DG XV : working group for caseins and caseinates

-meeting of the 10/9/74

e) Work in connection with the fertilizers group of I.S.O.

-October 1974 (Larisse-Haemers)

f) External activity : short consultation about the BCR technique from experts of U.N.I.C.H.I.M. (Associazione per l'unificazione nel settore dell'industria chimica - Milan); 3 and 4/10/74 (Larisse-Frangmeyer-Haemers).

1.4.3. Difficulties

The technical difficulties raised can be summarized in three point:

a) The statistical results have to be interpreted at each step of the calculus and discussed within the working groups. Due to the very large number of statistical analysis to be performed and the very small number of statisticians at disposal, it is obvious that, even if we use in all cases the Aspin-Welch technique, the different steps, and in particular, the last one, i.e. the certification procedure, cannot be followed as it would be. The best way to drop this difficulty is to explain, by means of a short seminar, the deep meaning of a statistical analysis and, in particular, of the certification procedure. This seminar is planned for the next months.

b) As we have said above, sometimes it could be interesting to use some further information on the distribution of the measurement errors. It is not foreseen to work systematically in this direction.

c) A very large number of theoretical statistical problems raised up during the first meeting of the working group "Statistical Principles". Some of them can be easily solved, the others need much more manpower.
Consequently, due to the actual possibilities in manpower it cannot be expected to face and to solve these difficulties in the next future. But according to our opinion, the most important and most profitable progress to be realized actually in the framework of the BCR statistical activities would be a more deep knowledge of the statistical concepts from the side of the group leaders.

1.4.4. Publications

-"Statistical Activities in BCR", Fangmeyer, Haemers, Larisse Euratom Report to be published

-"BCR Statistical Technique", Fangmeyer, Haemers, Larisse 7th Materials Symposium in Gaithersburg, Maryland, organized by the NBS presented by Girardi.

1.4.5. Further works

-Further applications and development of designs of experiments (in particular Tribology applications)

-Organization of a short seminar on basic statistical concepts useful for BCR

-Preparation of the next meeting of the "Statistical Principles" working group.

REFERENCES

10) ISP-1973-01, RM-1973-001

11) EUR/C-IS/385/74, f of June 1974

12) BCR Information, Vol. 0, Number 0, March 1974

13) "Record of the 3rd meeting of the ACPM", Ispra 24-25 June, 1974 doc. BCR/75/74, 18 July 1974

14) see G. Fattori, F. Astigiano CEE-JRC-Ispra, Program Description Series, "KWIC /Caronte, A. Package for multiple handling/editing of a list of information".

2. SUPPORT TO THE COMMISSION'S SERVICES

2.1. Analytical Methods for Pesticide Residues in Plants and Vegetables (Versino B.)

A final version of the EEC method for determining pesticide residues in plants and vegetables has been prepared in such a way that it summarizes all the consistent and inconsistent data obtained in the course of various interlaboratory analysis campaign.
A selection of analytical methods has been done which can be used as alternative to EEC method because their validity was checked through collaborative analyses by different laboratories. In other words the final goal is to provide a guidebook for the analysis of pesticide residues including some chapters dealing with general procedures (applicable to all the methods) and a series of analytical methods (presented in a standard form) each one with the pertinent application field.
The same criteria will be applied to the analytical methods actually under development for determining pesticide residues in animal foodstaff (milk, eggs, meat, etc.).
As far as the instrumental development is concerned, a paper has been presented at the IUPAC 3rd International Congress (Hensinki, July 3-9, 1974) on the optimisation of a selective gas-chromatographic detector (dual flame photometric detector) developed in JRC-Ispra laboratories in 1971. The features of this detector are fully described in a peper in press on "Chromatographia".

2.2. Analysis of Fertilizers (Serrini G.)

As a continuation of the activity described in 1973 year report, EEC and BSI methods for fertilizers analysis have been checked to ascertain the feasibility of adopting and diffusing them as ISO recommended procedures. To achieve this goal an interlaboratory campaign analysis has been launched for Nitrogen (nitrate form) determination in fertilizers with the cooperation of 9 laboratories (4 from United Kingdom, 4 from Italy in addition to the JRC-Ispra).
As far as the total nitrogen (13–22%) determination is concerned both methods showed to be equivalent, all the data being in the range of $\pm$ 1% related to the mean value. Detailed information are given in doc. ISO/TC/134/SC 4/WG 1 (CEE) 49, discussed in the course of the ISO meeting held in London on October 1974.
Four different analytical techniques have been employed for nitric nitrogen determination i.e. gravimetry, titrimetry with potentiometric end-point determination, spectrophotometry and selective electrodes. However the 2 electrochemical techniques have been considered only for test purposes. In the N_2 concentration range $< 2.5 - 10\%$ almost all the data were found to be scattered within $\pm 2\%$ relative to the general mean value. A close examination of the various interferences affecting the accuracy of the methods is actually in progress.

2.3. Analysis of Surfactants (Serrini G.)

With the aim of extending to non-ionic surfactants the directive already established for the ionic ones, the OECD working group (to which a JRC Ispra representative is joined) has reviewed all the methods presently available for determining the biodegradability of these products. These methods seem to be successfully applied to all but a restricted family (nonil phenol type) of surfactants and, apart from minor modifications, are similar to those included in the directive and valid for anionic surfactants.

2.4. Lead and Cadmium release from glazed ceramic tableware (Rossi G.)

In the course of 1974 extensive analytical work has been carried out for determining Lead and Cadmium release from ceramic tableware through the "cold test" procedure (24 hours leaching with 4% acetic acid at room temperature). Direct determination of Pb and Cd are currently performed on the Test solution by flame (for high concentration levels) and flameless (low concentration level) atomic absorption spectroscopy. As a result of the experience gained in this field technical support has been given to the working group "Elimination of Technical Obstacles to trade - Lead and Cadmium release from ceramic tableware" in the framework of the activity of General Direction XI/A/2, for the preparation of a draft proposal for directive on this subject.
Moreover the JRC-Ispra laboratories have been entrusted to study the feasibility of setting up a "hot test" to which over-proof ceramic tableware should be submitted or of establishing a relationship between the cold and the hot tests. This work will be done in the course of the year 1975.

2.5. Cosmetics (Elimination of technical obstacles to trade) (Sandroni S.)

In the framework of the directive on "Cosmetic Products" a secretariat support has been given to the working group "Analytical Methods" dealing both with chemical and biological methods. To this working group some 30 restricted subcommittees belong each one charged to develop a specific analytical method for a selected problem.
In the course of 1974 two meetings of the working group took place on May 21st-31st (8th meeting doc. XI/426,74) and on November 25th-27th (9th meeting, minutes not yet available). As a result an agreement has been reached unanimously on the following items:

- sampling procedures for aerosols
- analytical methods for Zn and thyophenate, oxalic acid and alkaly nitrites in cosmetic products

Two more meetings have been planned for 1975.

2.6. Classification, Labelling and Packaging of Dangerous Substances and Preparations (Mosselmans G.)

During 1974 the activities were concentrated on the following three branches of the sector:

-revision of the Council Directive 67/548 (5th amendment) and his annex

-preparation of the final proposal for the directive paints, varnishing, printing ink and similar products

-preparation of the final proposal for the directive pesticides

2.6.1. Council Directive n°67/548

This Directive regards the classification, the labelling and the packaging of dangerous pure substances. In agreement with the decision of the Council, the Committee for adaptation of directives to the technical progress was created. Afterwards the Committee has adopted his internal regulation.
The working group has decided to revise the annexes of the Directive.

Annex III: Nature of the special risks attaching to dangerous substances

Annex IV: Safety advice concerning dangerous chemical substances

Annex II : Symbol to use

Annex I : List of dangerous substances classified in the order of the atomic number of the element most characteristic of their properties.

Actual State of Works

-Revision of annex III and IV is finished. The final redaction is on hand.

-Due to the great difficulties for the harmonisation of the symbol at international level the working group has decided to change nothing at the actual annex II. He suggests the Commission to contact other international organisms (ISO-UNO) to study together harmonisation of symbols.

Contribution of the JRC-Ispra

Preparation and drafting of the technical annex following the conclusion of working group's discussions.

2.6.2. Paints and Varnishes

Actual State of Works

The final proposal of the Directive is being written and will be transmitted to the Council during February 1975.

Contribution

Draft of the technical annex (list of substances and definition of the limits of concentration which require the labelling of the preparation) based on the data given by Industry Representatives, the different characteristics of substances (chemical, physical and toxicological) and the conclusion of the different meetings of the working group.

2.6.3. Pesticides

Actual State of Works

The final proposal of Directive was submitted end of December to the Council.

Contributions

Draft of the technical annex (list of the substances and definition of the limits of concentration which require the labelling of the preparation). This was made following the same process that is used for paints and varnishes.

The JRC participation consists in preparing the documents which are required for writing the technical annexes (research of the characteristics of the substances used in the preparations countersigned by the Directive) and the final edition of those annexes sometimes with translation of the technical expressions.
To make these works, we have jointed 15 meetings.

2.7. Nomenclature of the Common Customs Tariff (Capobianchi S.)

A provisional inventory was printed in July 1974 with the help of the CETIS computer (doc. GUD/661/74).
It reproduces the organic-chemical demonimations of the various items appearing in the divisions and subdivisions of chapter 29 of the Joint Customs Tariff together with explanations.
It is composed of four single-language volumes (French, German, Italian and Norwegian) each one with a customs' classification for each product and one volume repeating the same denomination in the four languages.
The inventory in each language covers some 7.100 denominations (5600 products and 1500 synonims). On 25/10/1974 the Commission granted the permit for publishing this provisional document. After correction of a few mistakes, the final version got printed end of December 1974.
In liaison with the various services of headquarters, the opportunity was examined to link the present EEC listing system on chemical products with other listing systems (chemical abstracts amongst others).

Estimations for 1975

The external publication of the final document should take place in May 1975 and an addendum should be added end of 1975. It is also foreseen to add to this group the inorganic products and those of the Nimexe.
All activities for the management of the Customs' Union are carried out in close cooperation with the Luxembourg services, the U.K. public service and with the help of technical personnel from Petten.

2.8. Participation to the most important Meetings

B. Versino
D.G. VI - Pesticide residues 24-25 June 1974; 12 November 1974, Brussels

G. Serrini, G. Renaux
ISO working group "Analysis of fertilizers" 14-15 October 1974, London

G. Serrini
OECD - working group "Biodegradability of surfactants" 28-29 February; 4-5 July 1974 - Brussels

S. Sandroni
D.G. XI - Cosmetic Products 29-31 May; 25-27 November 1974, Brussels

G. Rossi
D.G. XI - Pb and Cd release frame ceramic tableware 25-27 September 1974, Brussels

G. Mosselmans
Yellow book: five meetings
Paints and varnishes: three meetings
Pesticides: seven meetings

3. REFERENCE MATERIALS AND CHEMICAL ANALYSIS (G. Rossi)

In the 1973's yearly report, the origin and the aim of the programme as well as the choice of the reference materials families, for which the work summarized in this paragraph has been carried out, have been described. Consequently this report deals with the progress accomplished in 1974 in the previously selected activities. However some new topics have been introduced for which introductory remarks are also included. The selection of these new projects is in general connected with the recommendations expressed by the various working groups or has been originated by the closeness with other research programmes under development in the J.R.C.-Ispra.

3.1. MR for Non-Ferrous commercial ores and electrolytic copper (in connection with the indirect action)

3.1.1. Pb, Sn, Zn and Cu ores

No further work has been carried-out in this field. In fact a preliminary assessment and statistical treatment of all the analytical data from the laboratories participating to the analysis campaign had to be done to ascertain whether a second round of analysis would be necessary or a preliminary certification could be issued. A meeting of all the participating laboratories being scheduled for the beginning of 1975, a decision on this matter will sonn be taken.

3.1.2. Electrolytic Copper

With the exception of the elements Zn and Sn, all the impurities considered for the analysis campaign have been analyzed by applying different analytical techniques. In particular :

Sulfur: as an alternate to the combustion followed by differential conductimetry, whose results were included in the 1973 report, spectrophotometric determination of H_2S produced by the dissolution of the sample, Sulphur oxydation to SO_4^{--} and subsequent reduction to S^{--} has been carried out. The mean value of 20 determinations (6 ppm $\pm$ 1.07 (σ)) is coincident with the previously reported result (6 ppm $\pm$ 0.54 (σ)).

Nickel: Ni determinations have been carried out both by flameless atomic absorption spectrometry and spectrophotometry after the separation of the base element by electrolysis. The material deposited on the anode has been dissolved and the solution added to the purified electrolized solution. 2.3 ppm of Ni (σ = 0.33) has been found, as a mean value of ten determinations by the first technique; 2 ppm (mean value of two determinations) were found by spectrophotometry.

Lead: the separation procedure described for Ni has been used also for Pb. The final determination by flameless atomic absorption spectrometry led to the value of 1.9 $\pm$ 0.13 ppm (σ) as a mean value of 8 determinations.

Antimony: two different analytical methods have been applied for Sb determination i.e.:

- chemical separation of Sb in hydrochloric solution by isopropyl ether and spectrophotometry of the chloroantimonite-rodamine complex;
- coprecipitation of antimony hydroxide together with other trace elements by means of sodium hydroxide using Lanthanum as carrier and flameless atomic absorption spectrometry.

The results obtained by the two techniques respectively 0.37 ± 0.08 ppm (10 determinations) and 0.45 ± 0.12 (5 determinations) are in close agreement with the previously reported data from nuclear activation analysis (0.436 ± 0.019).

Bismuth: the coprecipitation enrichment procedure used for Sb proved to be effective also for Bi. The standard addition method for the quantitative determination by flameless atomic absorption spectrometry led to a mean value of 0.53 ± 0.15 ppm Bi (5 determinations).

Silver: A new series of measurements has been carried out by nuclear activation through a chemical separation procedure (based on Dowex 1 x 8 ion exchange and SnO_2 inorganic exchange columns) and NaI (Tl) multichannel spectrometry. The mean value of 13 determinations (8.77 ± 0.3 ppm) is in fairly good agreement with the value (7.6 ± 0.56 ppm) given by flame Atomic Absorption Spectrometry. For this last technique no enrichment procedure or chemical separation has been found to be necessary.

Tellurium: 0.124 ± 0.014 ppm of Te have been determined as mean value of 14 measurements by nuclear activation analysis.

I^{131} from the Te^{131} and Te^{131m} decay is extracted with benzol from the sample solution after the addition of NH_4I as carrier.

Arsenic: As determinations have been carried out by nuclear activation analysis after the separation from the Cu base by means of a Dowex 50 x 8 ion exchange column. The mean value of 14 determinations (2.07 ± 0.12 ppm) is consistent with the previously reported value (1.8 ± 0.21 ppm) obtained by spectrophotometry. Exceedingly high blank values prevented to obtain consistent data by flame of flameless atomic absorption spectrometry.

3.2. Reference Materials for Non-Ferrous Metals (in connection with indirect action and Eurisotop activity)

3.2.1. Determination of gases in metals

Oxygen determination in non-ferrous metals

The results obtained in 1973 on Pb, Cu (oxygen-free and deoxidized) and Zr (500 and 1000 ppm) have been discussed at the meetings held in Brussels on March 5 and June 4-5, 1974, together with those from the other cooperating laboratories.
As a result a certificate for oxygen content could be issued for these samples. Detailed information on the analytical method used, on the analytical data and on the certification procedure are given in the

Eurisotop Office publication ITE 79, 80, 81 and 82.
In the course of 1974 main efforts have been directed towards the setting up of vacuum fusion methods for oxygen determination in W (Pt bath, 1900°C) industrial Cu (graphite crucible 1450°C) and Pb (graphite crucible, 1150-1450°C). In the case of industrial Pb the agreement among the results so far achieved is such that the establishment of a certificate can be reasonably planned for 1975. (Meeting held in Brussels on November 27, 1974).
A series of determinations have been carried out on industrial Cu by H_2 reduction. The value of 135 ± 3.1 ppm O as a mean of 20 determinations is in close agreement with that selected for the provisional certificate (138 ± 7 ppm).

Nitrogen determination in refractory metals

The Kjeldahl method has been applied for determining nitrogen in W and Ta. As the mean value of 10 determinations 4.8 ± 1.5 and 11.0 ± 2 ppm has been found for the two metals respectively. However taking into account the very large discrepancies existing between the analytical data of the cooperating laboratories a general revision of the whole programme involving the development of new methods has been decided. As a consequence the Kjeldahl method has been temporarily discontinued because of the low concentration of N in these materials and of the difficulties associated with the dissolution of the sample. Tests have been carried out by vacuum fusion on W in different environmental conditions (Pt bath, 1900°C, Pd flux and Ni flux, 1650°C) with the aim of achieving the complete release of nitrogen. The accuracy achievable at present can be considered satisfactory for industrial purposes.

3.2.2. Determination of noble metals in Cu

As a further development of the programme on the assay of traces of noble metals, four Cu samples containing respectively 12 and 1.2 ppm Pt and 12 and 1.2 ppm Pd (nominal values) have been prepared by BCMN-Geel and distributed to all the cooperating laboratories for the analysis.
As far as the JRC Ispra laboratories are concerned flameless Atomic Absorption Spectroscopy and nuclear activation analysis have been applied using the chemical separation procedures described in the 1973 report. The experimental results, which will be discussed in a meeting to be held on February 17th, 1975, are collected in the following table (mean value of 6 determinations).

Sample		FAAS		NAA	
Pd 12 ppm	11.8	± 0.99	12.3	± 1.06	12.35 ± 0.3
Pd 1.2 "	1.25	± 0.09	1.17	± 0.09	1.19 ± 0.08
Pt 12 "	11.83	± 0.13	11.50	± 0.65	12.00 ± 0.61
Pt 1.2 "	1.22	± 0.05	1.13	± 0.1	1.43 ± 0.11

Moreover the 1000 ppm Pd and 100 ppm Pt samples have been analyzed by nuclear activation analysis. The value of 93.67 ppm Pd and 95.41 ppm Pt which have been found fall well in the range covered by the cooperating laboratories (99.86 ± 10 and 99.38 ± 10 respectively) and are in close agreement with the previously reported Flameless atomic absorption data.

3.3. Reference Materials for Ferrous Metallurgical Products

(in connection with indirect action)

3.3.1. Reference Materials for Coke Powder

On the basis of the recommendations formulated by the working group "RM for metallurgical ferrous products" (7th meeting January 9th 1974, doc. Eur/C-Is/104/74) an analytical intercomparison campaign having the aim of certifying the composition of four coke samples has been launched, the JRC-Ispra laboratories being included among the cooperating laboratories. The samples have been prepared by the British Carbonization Research Association-Wingerworth U.K. under the supervision of Dr. H.C. Wilkinson, and distributed to some 25 european laboratories including JRC-Ispra.
The various parameters to be certified, i.e. moisture, ash, carbon, hydrogen, nitrogen, sulphur, chlorine, phosphorus in ash, have been determined in the JRC laboratories as requested, through widely accepted and well defined analytical procedures (ISO R 687, ISO R 1171 and ASTM D 271 methods). Table I collects the pertinent analytical results so far obtained.
A preliminary discussion of the results of this analysis campaign will presumably take place early in Spring 1975, when all the data from other cooperating laboratories will be on hand.

3.3.2. Reference Material for Coal Samples

With the secretariat support of the JRC-Ispra a project is currently under study by the working group for the preparation of reference materials for coal (minutes of the 9th meeting, May 15th 1974, doc. AC/cf-00800/3393,74). As soon as stability test will be completed (actually in progress at Ruhr Kohle A.G.-Essen) a decision will be taken on the parameters to be determined and certified. Major and trace elements determinations both in coal and ashes can be reasonably scheduled to be required in 1975. To this scope the work actually in progress at JRC on NBS coal samples (see under 3.6.2) can be considered of great help from the analytical point of view.

3.4. Reference Materials for Inorganic Chemical Products

3.4.1. Reference Materials for Organo-Metals

Since the general inquiry on the needs of reference materials, carried-out in 1972, had shown the interest of some laboratories for reference samples for determining inorganic impurities and additives

in petroleum products, a more exhaustive inquiry has been carried-out among the 17 laboratories who expressed needs for new or better reference materials.
As a result only 6 laboratories gave an answer and among these 5 claimed to have no more problems concerning the metallo-organic compounds. The remaining laboratory stressed some difficulties associated with the unstability of the NBS Si compound, when diluted at very low concentration, and with the solubility of the NBS Fe and V compounds.
Taking into account that at the present a large choice exists of commercially available metallo-organic compounds (about 40 elements) and of several series of blended standards for determining trace metals in oil, one can conclude that it would be irrealistic to start with work in this field. Possibly, stability limitation of these compounds particularly at low concentrations, together with compatibility and solubility studies could represent the main objective.

3.4.2. Preparation and Characterization of High Purity Acids

In connection with the recommendation given by the working group "RM for inorganic chemical products" (minutes of the 6th meeting, February 21st, 1974, GR/cf-00800/3153), feasibility studies have been started with the aim to produce, on a laboratory scale, high purity mineral acids by the sub-boiling distillation technique.
Preliminary tests are being carried-out on HNO_3 using quartz vessels. Flameless atomic absorption spectrometry is planned to be mostly used for quantitative determination of trace metals.

3.5. Reference Materials for Environmental Pollution Studies

3.5.1. Reference Materials for Airborne Particulates

As a consequence of the programme started in 1972 for the development of analytical technique for the quantitative determination of environmental pollutants, an interlaboratory comparison analysis has been launched on a batch of air particulates (about 600 gr) collected during winters of various years from 1962 to 1972 by the Provincial Laboratory for Air Pollution of Milan.
The aim of this campaign, to which some 38 european and american laboratories have participated, was mainly to characterize the sample as fully as possibly, to evaluate both the potentialities and the accuracy of the various analytical techniques, and to ascertain the feasibility of certifying the chemical composition partially or totally in such a way to use this sample as reference material for further application in the same fields.
In table II are listed the analytical techniques used by the various laboratories. As far as the JRC-Ispra laboratories are concerned, nuclear activation, X-ray fluorescence, flame and flameless atomic absorption, emission spectroscopy, flame photometry, combustion techniques and wet chemical methods have been employed for the most of the elements listed in table III.

A meeting has been held in Ispra on July 10th, 1974 with the participation of most of the cooperating laboratories to discuss the analytical data obtained.
As a result of this meeting the following conclusions can be drawn:

1) A good agreement has been found by the various laboratories using different analytical techniques for the elements Ca, Mn, Fe, Na, and Zn.

2) The data on some 10 elements (including the most significant from ecological point of view As, Al, Cu, C, Sb and Hg) look rather promising and a further agreement can be reasonably expected through a refinement of the analytical methods.

3) Doubtful results have been presented for the elements Ni, V, Pb, S, F, Ti, Cd, Cr and Sn. For these elements deep revision of the analytical procedures must be carried-out.

4) The feasibility of preparing a RM for environmental purposes seems to be ascertained and further interlaboratory comparison analysis on this as well as on other samples is recommended.

3.5.2. Characterization of Mercury Compounds

In connection with environmental problems, mass spectrometric studies have been carried-out on mercury organo-compounds whose importance does not need to be overstressed. To achieve this goal a series of simetric and asimetric mercury compounds has been synthetised namely: dimethyl mercury, diethyl mercury, di-n-propyl mercury, di-n-butyl mercury, di-isobutyl mercury, di-n-amyl mercury, di-p-anisyl mercury, di-p-chlorophenyl mercury, di-p-tolyl mercury, di-p-nitrophenyl mercury, di-o-tolyl mercury and diphenylethyl mercury. For this first six compounds mass spectra at various excitation conditions have been obtained in view of their qualitative and quantitative determination.

3.6. Cooperation with the National Bureau of Standards

Within the framework of this cooperation whose scope has been discussed in 1973 report, the following progress has been accomplished.

3.6.1. Determination of Major and Trace Elements in Bovine Liver

The work on this SRM has been completed. Ca determinations have been carried-out by flame atomic absorption spectroscopy using 10% La as releasing element to suppress P interferences. The obtained value of 128.3 ± 6.8 ppm is in close agreement with both the previously reported nuclear activation value (125 ppm) and the NBS preliminary data (123 ppm).

Direct determination of Ag on the solid sample have been performed by flameless atomic absorption spectroscopy using aqueous solutions as reference standards and the standard addition technique. The value of 0.087 ppm thus obtained is somewhat higher than the preliminary value given by NBS (0.065 ppm). On the other hand the dissolution of the sample led to exceedingly high blank values preventing to obtain consistent data.

3.6.2. Analysis of Coal Samples

The work on SRM 1631 (sulphur in coal) has been completed and all data forwarded to NBS. A new certificate for the 3 samples (A, B, C) has been issued by NBS including the analytical data obtained in the JRC Ispra laboratories.
As supplementary information Pb and Hg determinations carried-out on the first two samples (A and B) have been also included in the certificate (see Annex IV).
Sulphur determinations have been carried-out also by neutron activation analysis (Cerenkov counting of Pb ^{32} formed by the neutron S^{32} (n, p) $\longrightarrow$ P^{32}) after a separation step on an acidic Al_2O_3 column. However apart from sample A, results somewhat lower than the Escka combustion technique have been obtained.
Consequently these data could not be used for certification purposes.
Preliminary determinations have been carried-out on SRM 1632 (trace metals in coals) by neutron activation analysis (Ge, (Li) multichannel spectrometry) and by X-ray fluorescence analysis (synthetic mixture as reference standard) with the following results (concentration expressed as ppm where not indicated).

Element / Method	Co	Cs	Fe	Rb	Sc	Ta	Tb	Th
N.A.A.	5.9	1.65	0.85%	24	4.3	0.26	0.22	3.05

Element / Method	Zn	Ni	Cu	Mn	Cr
X.R.F.	13	3	$\lesssim$2	19	$\lesssim$1

The application of flame and flameless atomic absorption spectroscopy is currently in progress.

3.6.3. Analysis of Vegetable Samples (Citrus Leaves, Tomato Leaves, Pine Needles, Alfaalfa)

Homogeneity studies are being carried-out on these sample using nuclear activation analysis, X-ray fluorescence and flame atomic absorption spectroscopy.

Reproducibility tests have been completed for K and Mn by X-ray fluorescence although not yet evaluated. Nuclear activation measurements on Cl, K and Mn and atomic absorption measurements on Mn at different sample size (100-300 mg) are planned to be completed early in Spring 1975. The determination of the various elements will be started as soon as homogeneity of the sample will warrant for consistent data.

PUBLICATIONS

A. Colombo
"Vacuum fusion aluminum bath method for the determination of hydrogen in copper"
Anal. Chim. Acta 72, 401 (1974)

F. Girardi
"Accuracy of chemical analysis of airborne particulate results of an intercomparison exercise"
Paper presented at the symposium "Accuracy in trace analysis" NBS-Washington 7-11/10/1974.

G. Guzzi, R. Pietra, E. Sabbioni
"Determination of 25 elements in biological standard reference materials by neutron activation analysis"
EUR Report in press

G. Guzzi, F. Girardi, R. Pietra
"Automated radiochemical separations, counting and data handling procedures in the analysis of biological specimens"
Paper presented at the meeting on the "Determination of radionuclides in environmental samples and biological materials" Rome 15-17/10/1974.

TABLE I - ANALYTICAL RESULTS ON 4 COKE SAMPLES

Parameter	Sample	Air dried basis	Dry basis	Dry Ash-free basis	Test method
Moisture	1 2 3 4	0.03 % 2.23 " 0.42 " 1.78 "	/	/	ISO R 687
Ash	1 2 3 4	6.87 % 6.41 " 5.43 " 5.28 "	6.87 % 6.56 " 5.45 " 5.38 "	/	ISO R 1171
Volatile matter	1 2 3 4	/	/	/	/
Carbon	1 2 3 4	91.10% 86.72" 92.22" 89.54"	91.08% 88.70" 92.61" 91.16"	97.80% 94.93" 97.95" 96.34"	ASTM D 271
Hydrogen	1 2 3 4	0.15% 0.94" 0.35" 0.84"	0.15% 0.96" 0.35" 0.86"	0.16% 1.03" 0.37" 0.91"	ASTM D 271
Nitrogen	1 2 3 4	0.96% 1.66" 1.18" 1.22"	0.96% 1.70" 1.19" 1.24"	1.03% 1.82" 1.26" 1.31"	ASTM D 271
Sulphur	1 2 3 4	1.29% 1.46" 0.51" 0.50"	1.29% 1.49" 0.51" 0.51"	1.39% 1.59" 0.54" 0.54"	ASTM D 271
Chlorine	1 2 3 4	< 0.004% 0.007 " < 0.004 " 0.005 "	< 0.004% 0.007" < 0.004" 0.005"	< 0.004% 0.008" < 0.004" 0.005	ASTM D 271
Oxygen	1 2 3 4	/	/	/	/
Phosphorus Ash	1 2 3 4	0.018% 0.014" 0.004" 0.003"	0.018% 0.014" 0.004" 0.003"	0.019% 0.015" 0.004" 0.003"	ASTM D 271

TABLE II

AIRBORNE PARTICULATES ANALYSES

ANALYTICAL TECHNIQUES

LAB	NA	AA	XF	ES	CA	CO	LI	OR
1				■				
2	■							
3			■		■			
4	■	■				■		■
5	■							
6		■						
7	■							
8		■						
9								■
10					■	■		
11	■	■			■			
12		■			■	■		■
13	■							
14		■			■			■
15								■
16	■							
17		■	■	■		■		
18	■	■	■	■	■	■		
19	■	■	■	■	■	■		■
20	■							
21	■	■	■					
22			■					
23		■						
24								■
25								
26	■						■	
27								
28								
29								
30		■						
31								
32		■			■	■		■
33								
34								
35								
36								■
37								
38			■					■

NA. NEUTRON ACTIVATION ANALYSIS
AA. ATOMIC ABSORPTION AND FLAME PHOTOMETRY
XF. X RAY FLUORESCENCE
ES. EMISSION SPECTROMETRY
CA. WET CHEMICAL TREATMENTS WITH FINAL GRAVIMETRIC, VOLUMETRIC, ELECTROCHEMICAL OR SPECTROPHOTOMETRIC MEASUREMENTS
CO. COMBUSTION METHODS
LI. ACTIVATION ANALYSIS WITH LINAC
OR. ORGANIC ANALYSES

TABLE III

Mean Values Obtained by Different Techniques - The First Figure in Brackets is the Number of Contributing Laboratories, the Second One is σ % - When the Mean Value is in Brackets the σ % Exceeded 100%

Element	Nuclear Activation	Atomic Absorption	Emission Spectroscopy	X-Ray Fluorescence	Wet Chemical Analysis	LINAC Activation Analysis	Combustion
Ag	15 (7-23)	9.7 (1-)	6 (1-)				
Al	16960 (8- 6)	13500 (1-)	18556 (3-10)		15120 (3-39)		
As	92.8 (7-10)			116 (1-)		116 (1-)	
Au	2.6 (8- 9)						
Ba	1052 (6-16)	558 (1-)	409 (2-80)	809 (2- 2)			
Br	624 (9- 9)			682 (2-30)		752 (1-)	
Ca	43160 (5-13)	38360 (8-16)	37500 (1-)	37060 (5-24)	37940 (2- 2)		
Cd	44.2 (1-)	58 (8-65)	48 (1-)	91 (1-)	73 (1-)		
Ce	150 (8- 6)						
Cl	11480 (9-12)			11500 (1-)	10500 (1-)		
Co	17.5 (10-22)			65.7 (1-)			
Cr	200 (10-15)	253 (6-73)	(333) (3-)	(2520) (3-)	125 (1-)		
Cs	2.5 (6-22)						
Cu	652 (3- 7)	697 (9-14)	648 (3-15)	(845) (4-)	647 (1-)		
Dy	2.6 (1-)						
Eu	0.5 (3- 9)						
F	2428 (1-)				2330 (1-)		
Fe	35040 (1- 6)	32256 (8-12)	32330 (3- 8)	32220 (4-14)	37000 (2- 8)		
Ga	10.7 (1-)						
Hf	1.3 (4-10)						
Hg	23.2 (5-59)	16.5 (2- 1)					
I	14.3 (1-)					14.2 (1-)	
In	0.5 (2-34)						
K	7540 (8-14)	6596 (5-25)		6800 (1-)	14200 (1-)		
La	83 (7-10)						
Lu	0.17 (2-29)						
Mg	6108 (2-38)	7332 (7- 8)	6183 (3-50)		8000 (2-11)		
Mn	1274 (10- 6)	1290 (8-21)	1528 (4-17)	1695 (5-36)	1080 (1-)		
Mo	31.8 (1-)		(157) (2-)				
Na	5904 (10- 8)	9240 (5-20)	13200 (1-)		19400 (1-)		
Nd	(65) (1-)						
Ni	1410 (5-27)	1517 (7-15)	1824 (4-35)	(2971) (5-)		1540 (1-)	
P			1400 (1-)		1445 (2-25)		
Pb		5540 (14-28)	5924 (4-12)	8050 (5-41)	8766 (1-)	6172 (1-)	
Rb	39.3 (1-)					38.5 (1-)	
S	39200 (1-)		44600 (2- 6)	43000 (1-)	45950 (2-20)		47800 (1-)
Sb	283 (11-13)			296 (1-)			
Sc	3.2 (7- 7)						
Se	10.3 (5-15)						
Si	61600 (2- 2)	75300 (1-)	62100 (2- 9)	78000 (1-)	55900 (2- 4)		
Sm	7.8 (5- 4)						
Sn		3100 (1-)	(333) (3-)	175 (1-)			
Ta	1 (1-)						
Tb	0.73 (1-)						
Th	3.4 (9-21)						
Ti	2715 (5-10)		2163 (3-21)	2466 (3-24)	2550 (2- 3)	3094 (1-)	
U	1.15 (1-)						
V	1990 (9-17)	2952 (1-)	1796 (3-34)	2710 (3-44)			
W	16.7 (4- 9)						
Yb	0.84 (1-)						
Zn	7434 (9-14)	7461 (9-12)	5747 (3-67)	8978 (1-)	9100 (1-)		
Zr						56.6 (1-)	
C							423150 (6- 4)
H					16500 (2- 4)		16560 (1-)
N							21000 (3-13)
O	279000 (1-)						144000 (1-)

4. REFERENCE MATERIALS AND PHYSICAL PROPERTIES

4.1. Thermal Conductivity (E. Aranovitch)

The 1974 activity which has been essentially a continuation of the work started in 1973 has concentrated on the study of a reference method for solid materials of medium and low conductivity, on the acquisition of high-standard measuring chains, on the setting of an apparatus for measuring the conductivity of liquids and on the study of heat transfer phenomena in fibrous structures.

4.1.1. Materials of medium and low Conductivity Materials

The study of a transient method based on the hot wire principle, using the inflexion point in the temperature profile has been continued in the temperature range from ambient temperature to 1300°C. A systematic study of sources of errors has been made. Improvements have been carried-out in the measuring chain with an automatic data acquisition system, in the stability of the heat source and of general temperature conditions. The method has been extended to various materials such as refractories, glasses, powders, sands, cement. It is particularly suited for wet cement where steady state methods cannot be used. Measurements have been performed on a glass fiber. used as a reference material on which various external laboratories are also making measurements with different transient and steady state methods.

4.1.2. Liquids

An apparatus for the measurement of the thermal conductivity of fluids has been mounted and preliminary calibration measurements have been made. This apparatus is based on a steady state method using a plane geometry. In order to obtain a greater temperature stability, the cold plate is maintained at uniform temperature by a two-phase fluid ambiance controlled by pressure. Difficulties had to be overcome in the mounting of the heating system. This apparatus will be tested with different reference liquids. The measurement of the thermal conductivity of liquid is of special interest in the petrochemical industry.

4.1.3. Fibrous Structures

The use of fibrous structures is becoming very important in the field insolations but up to this date there exists no agreed upon method to measure with precision the apparent global thermal conductivity of such structures because of the complex modes of heat transfer by conduction, radiation and natural convection. An experimental programme has been started to study the influence of various parameters such as apparent density, pressure, temperature gradient, ambient gas on the global conductivity. A great deal of further investigation will be necessary before clear conclusions can be obtained.

Two reports are being prepared on point 4.1.1 and 4.1.2. At the end of 1973 a paper was presented at an International Conference on Thermal Conductivity in the United States.
The different activities, which have been mentioned, are closely connected to the indirect actions of BCR and active collaboration is planned with these indirect actions in the form of participation to interlaboratory comparisons.
An eventual collaboration with ASTM will be discussed at an ASTM Conference in London in May 1975.
Next year activity will concentrate on high temperature measurements.

4.2. Viscosity (G. Kuhlbörsch)

4.2.1. Absolute viscosimeter

The first absolute viscometer, built in 1936 by S. Erk and A. Schmidt, mainly served to determine the viscosity of water at 20°C with the highest precision obtainable as a base for the viscosity scale.
The value of viscosity of a substance at 20°C is, in many cases, considered insufficient today. Materials, whose viscosity values are available at various higher temperatures, are very much in demand. However, with growing Δt between room and measuring temperatures we must expect increasing difficulties in maintaining the required consistency of a temperature of 0,01°C during measurements, if we continue using the classical thermostat.
An absolute viscometer equipped with a heat pipe as thermostat is definitely superior to all other instruments. The temperature of the heat pipe may be regulated with the utmost precision via the gas pressure. It can hardly be said to be prone to disturbances. The viscometer furnishes precise data which make corrections unnecessary.
In its present form, the way it was constructed by S. Erk and A. Schmidt, the absolute viscometer may not be fitted into a heat pipe. It had to be re-designed without changing its principles. In addition to that, deficiencies of the old instrument were to be avoided.
In order to build such a viscometer four partial problems had to be solved first:

1) It is possible to construct a heat pipe large enough to house the entire viscometer? How constant a temperature can it maintain?

2) Can we find capillaries with an inner radius remaining constant throughout the entire length?

3) Can we find a system which permits the liquids to be measured to be forced through the capillaries at constant pressure?

4) Can the electronic accessories for temperature control, and for time- and pressure controls be procured at reasonable expense?

The results of our endeavours were the following:

1) The proto-type of a heat pipe got ready in spring 1974 in cooperation with the group "High Temperature Technology". While testing the appliance, we noticed that our expectations regarding temperature

constancy and precision were not only met, but even surpassed. The heat pipe, being at first only meant for a temperature of 250°C, was only fueled with water. However, water can only be expected to work well after it reaches a temperature of 40°C. We therefore have started an additional series of tests on the basis of a different working fluid in order to study the behaviour of the heat pipe at room temperature.

2) The diameter of capillaries, appearing in the Hagenpoiseuille equation at the fourth power may only feature an uncertainty factor of 0.1%. Hence, tubes of such precision may only be found when hand-picked by the manufacturer from a running production. Everyone of the firms we officially approached refused to do this. Thus, a whole year was lost before we obtained the capillaries via PTB. Four of these tubes with the inner diameter of 0.5, 1, 2 and 3 mm were thoroughly checked and measured at BCMN-Geel by Messrs J. Brulmans, F. Hendrickx and J. Verdonk. The measurements resulted in the following deviations:

capillary	0.5 mm	inner Ø	deviation from mean Ø	- 0.0001 mm
	1 "	"	" "	- 0.0001 mm
	2 "	"	" "	- 0.0002 mm
	3 "	"	" "	- 0.0005 mm

i.e. the deviation from the mean diameter is smaller than 0.05% in all capillaries, and thus even better than we asked for. These capillary tubes must now be divided in such a way, that pairs of equal inner diameter of 10 and 20 cm may be obtained. The end faces of the capillaries must be treated with the utmost care. The optical grinding must result in an inner rim of such smoothness, that any possible scratches remain smaller than the divergence from the mean radius.
While looking around for an optical firm to perform this task for us, we ran into some unexpected difficulties. Only towards the end of November 1974 did we find a firm ready to perform this special order. Delivery should, however, not be expected before spring 1975.

3) S. Erk and A. Schmidt were using a closed cylinder with suspended liquid column as their supply container. Its bottom was at an even height with the capillaries (Fig. 1). In such a system the hydro-static pressure may be eliminated, being solely dependent from the difference of height between the lower end of the gas input tube and the upper rim of the outflow tube of the cylinder. Thus, the pressure forcing the liquid through the capillaries corresponded to the given gas pressure. The latter was adjusted by immersing an open tube into a water column. However, according to Dr. Weber (PTB), irregular pressure changes were observed when using this system. These impeded the reading of the height levels in the ascending tubes.
One other reason why we could not adopt the system was, that in order to fit the system in the heat pipe thermostat, we would have to fix the supply container under the capillaries. For this reason we first attempted to solve the problem by using pumps. However, the resulting friction heat in the bearings proved to be too disturbing.

But since all mechanical solutions are suffering from that handicap, we returned to gas-pressure control. (Fig. 2). Owing to a higher pressure in the first chamber, the surplus liquid flows into a small overflow funnel in the second chamber. As there is a certain gas pressure even in this chamber, the liquid is forced into the capillaries. The only reason for the overflow funnel is to maintain an even level at all times, thereby preventing a pressure change caused by a diminishing liquid level in the supply container. The dynamic pressure of a liquid column is serving, once again, as pressure regulator. Pressure changes of the above mentioned nature were not observed. Their cause (creation of small pressure thrusts caused by the bubbles) could be eliminated.
However, one thing was rather cumbersome in this system: to stop the flow of liquid from the first to the second chamber.
Owing to the large diameter of the tube there was no clear-cut relation between pressure and regular flow quantity. This problem may be solved by using a special capillary (exchangeable). The mathematical relation is once again based on Hagen-Poiseuille's law.
As the viscosity has to be known at least approximately, the required pressure is easily determined with a desired flow. The capillary holder and the overflow funnel etc. have already been constructed as original parts. Besides, new regulating valves were ordered, permitting a fast and reliable precision regulation of the pressure.
The next step on the agenda is a thorough test of the original parts as to their proper functions. This is one of the most important items in the entire system.

4) The absolute temperature has to be measured in the supply container, as well as in the capillaries. Due to the required precision we need a rather costly measuring system. As temperature sensors we are using two standardized (Pt) resistance thermometers (transfer-standard). A "Universal Ratio Set" (Seven-decaded-precision-voltage-devider by Leeds & Northrup) was ordered for resistance measurements, enabling us together with the high precision appliances already at hand, to achieve the required precision. It is true that the compensator circuit requires more effort from us, but on the other hand the purchase of a special precision bridge could be avoided. The latter would have cost us 10 to 15000, i.e. about double or triple the price of the voltage devider. A further advantage of the above instrument is that it enables us to calibrate our digital voltmeter. The latter is used, together with a recording instrument, to perform a continuous checking on the differential temperature between the two temprature sensors. This is a safety measure we think indispensable. Owing to the fact that the absolute temperature can ly be periodically read manually, it could happen that a temperature disturbance between checks would slip by unnoticed, without constant controls.
Another additional appliance presently under construction is the electronic time keeper. In order not to make any mistake in time, the beginning as well as the end of a measurement must be made to coincide fairly precisely with the moment a drop drips down from the overflow tube. Visual time keeping was proven unsatisfactory in the case of fast dripping. The electronic time keeper lets the

droplets fall through a light beam, which throws its light on a photo diod, starting or stopping a quartz clock with the impulse resulting from such action. Also connected there is a system regulating the closure of the receptacle.
Another task received priority due to our participation in the indirect action. As we stated initially, the superiority of an absolute viscometer manifest itself only with the determination of higher temperatures. For this reason our plan provided also for measurements with Ubbelhode-Master viscometers in the 20°C region as routine and control measures. As we are required to participate as soon as possible in the ring measurements of the indirect action, we have started to assemble the necessary equipment. The newly acquired thermostat of the system was supposed to have a constant temperature of $\pm$ 0.01°C according to specification. But despite the already electronically performed regulations, such values were unobtainable. This was only possible after we inserted a proportional regulator of our own construction, which we had on hand from previous tests.
However, Dr. Smit (TNO) and Dr. Weber (PTB) were sceptical on account of the water volumes we used. It is their experience that a reasonable safety in temperature constancy can only be assumed when temperature disturbances may be evened out by sufficiently large water volumes. They reported they were using thermostats of their own construction of 250 to 300 liters capacity. Following their advise we ordered the construction of such a thermostat vessel.
The foremost task for the coming year, however, must be a scale of viscosities of our own, such as is required for the participation in ring analyses.

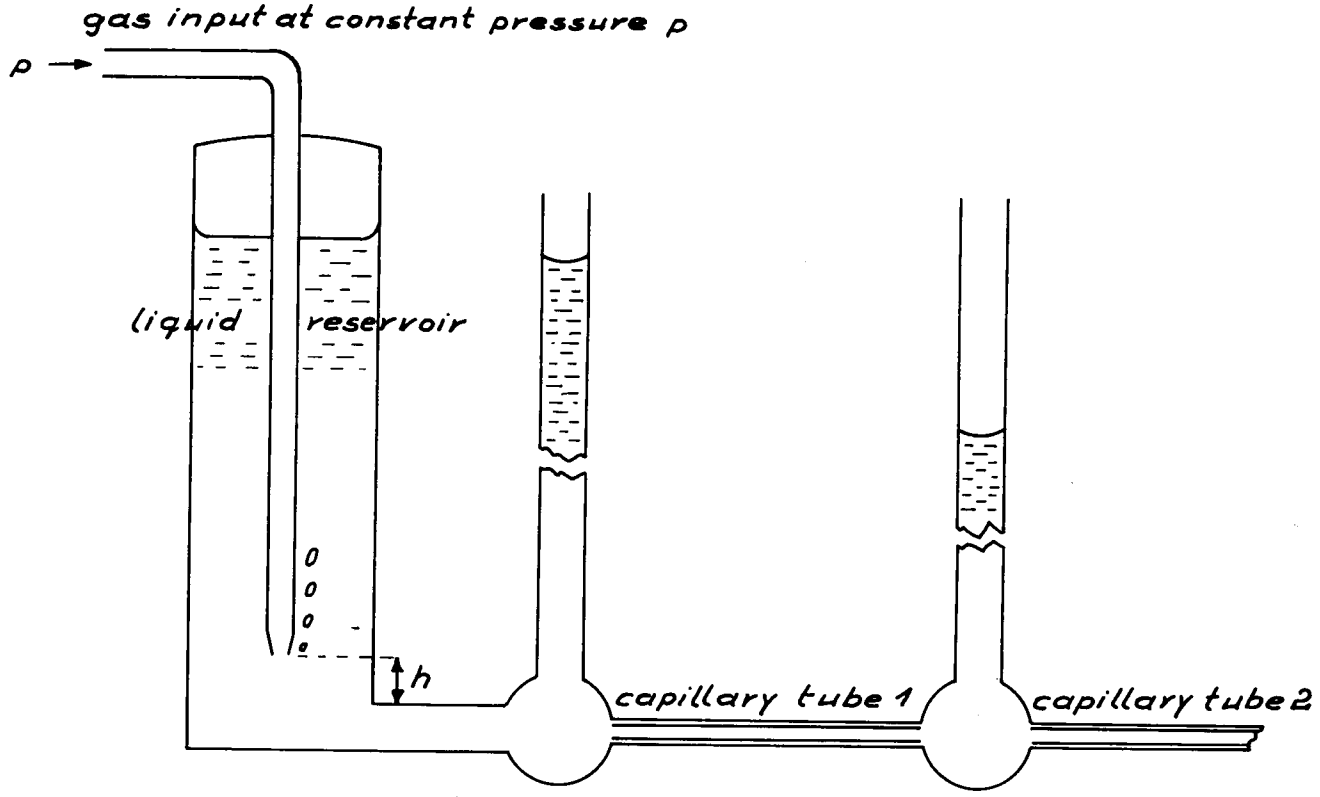
4.2.2. Heat-Pipe Thermostat (Bassani)

An heat pipe thermostat, of high thermal uniformity has been realized for viscosity measurements.
Temperature variations lower than $\pm$ 0.005°C have been performed in the inner chamber of the thermostat whose diameter is 20 cm and length 50 cm. These measurements have been realized for temperatures between 80°C and 160°C.
During the year 1975, it is foreseen to extend measurements to lower temperatures (20-80°C) and to realize an automatic control system of the thermostat.

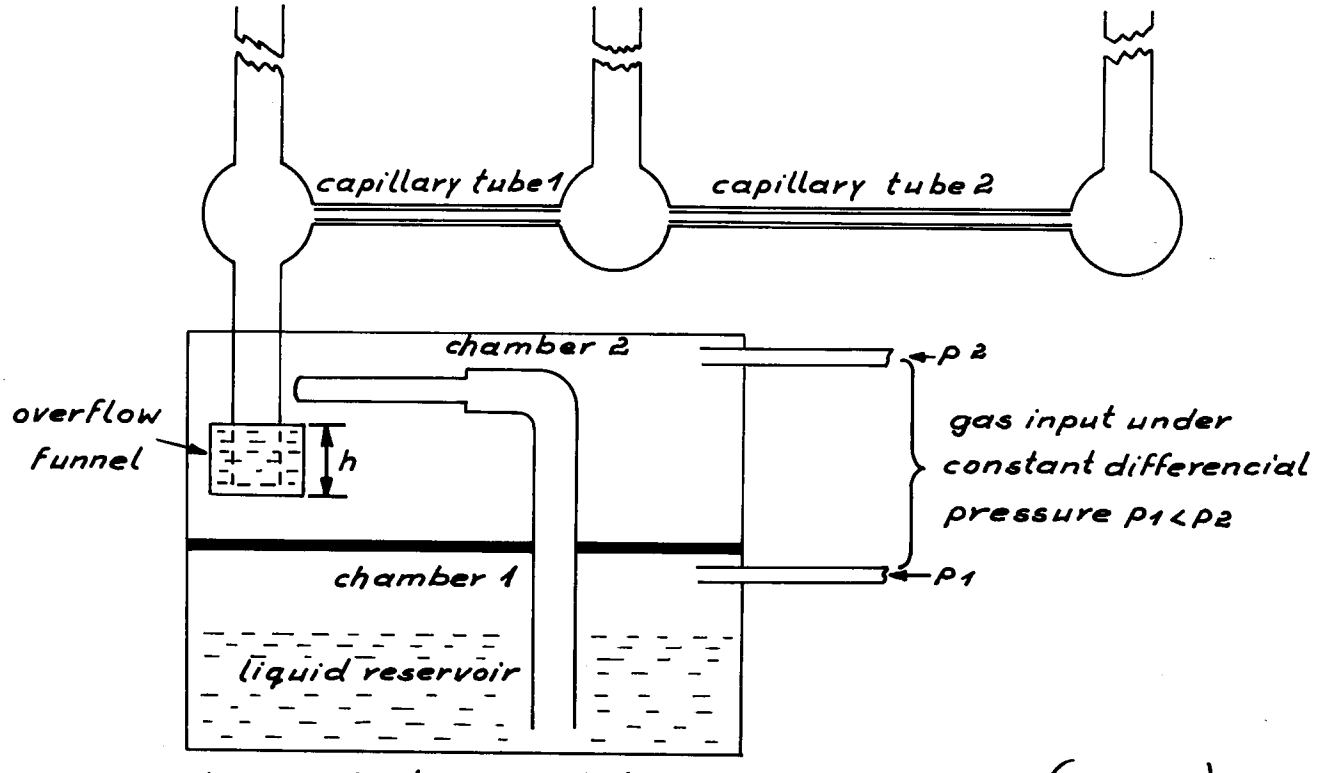
4.2.3. Indirect Action

Dealing with viscosity reference materials, it is impossible to continue manufacturing in the same way as was done with other reference materials. The short-life of the substances prevents us from keeping large stocks on hand and getting them certified by several different institutions. If we want to change the present, most unsatisfactory situation, we have to tackle the problem in some other way.
We therefore devised a plan providing a unified preconditioning and stocking, as well as equal origin of basic substances for all European suppliers of viscosity reference materials. The TNO and the PTB being leaders in this particular area, we contracted Dr. Smit and Dr. Weber last Spring, so as to acquait them with our plan. They were both convinced of the efficacy of the planned procedure. They promised to investigate and examine technical details. At a meeting of specialists in Ispra last September, Mr. Daborn

too, the representative of the NPL, was favourably impressed with the plan.
The certification of the reference materials being the sole responsibility of the supplying institutes, it is of the utmost importance that precision of measuring devices is in agreement between the various suppliers. This, at least, is the goal of the pending ring-analysis, for which the technical pre-conditions were devised at the same meeting in September.


gas input at constant pressure p
p
liquid reservoir
h
capillary tube 1
capillary tube 2
h = constant hydrostatic pressure

(Fig. 1)


capillary tube 1
capillary tube 2
chamber 2
p 2
overflow funnel
h
gas input under constant differencial pressure p1 < p2
chamber 1
p1
liquid reservoir
h = constant hydrostatic pressure

(Fig. 2)

5. REFERENCE MATERIALS AND TECHNOLOGICAL PROPERTIES

5.1. Tribology (B. Causse D'Agraives)

It is recalled that the Ispra team is mainly concerned with the different ways to get an acceptable and reproductible methodology for friction experiments. Contacts established with industries and laboratories have shown a general interest in developing a programme on the reproducibility of friction experiments with the further aim of producing calibrated friction samples and/or reference methods.
In 1974, the work has been the continuation of the 1973 participation in the direct and indirect action.
The main topics are:

- Development of a reliable double tribo-testing machine, in an attempt to estimate the influence of accuracy of the apparatus on the reproducibility of the results.
- Organisation of an intercomparison programme of friction measurements which involves at present three european laboratories.
- Performing friction measurements in hostile environments.

5.1.1. Double tribo-testing machine

The double tribometer, consisting of two coupled units , each one being a simple "pin-on-disc" pendular tribometer, has been already described (1) and (2). Using this apparatus, developed at Ispra, an attempt is made to compare two machines which are considered "identical". Furthermore, when the discrepancies between the results obtained with the two apparatus will fall within acceptable limits and be reproducible, one of the two units will be circulated in different laboratories in order to observe the reproducibility of the method in different hands.

Using accurately prepared batches of samples, consisting of flat sliders and discs made both from two different steels, iron and bronze (see (2)), we have performed series of experiments using simultaneously the two rigs which are driven exactly at the same speed by one single motor.

As the method for preparing the samples has shown good reproducibility (checks on surface finish and flatness), we were expecting to find a good similitude in the friction records of the two units.
It appeared, nevertheless, that some relevant differences remained in the results whatever the chosen samples pair was.

The test sequence was always the same (fig.3)that is:

- dry conditions, room temperature,
- two revolutions at low speed: 2.2 mm/s,
- a "running in" at 1,500 revs. at 220 mm/s (no friction measurements)
- two revolutions at low speed : 2.2 mm/s

Fig. 4 shows schematically the two coupled units seen from above, the pendulum beam, which is vertical at rest, not being visible entirely. The main parts are:

1) Main reclinable frame bearing the disc axle and 3 rigid columns (2 on fig. 3) to guide the counter-frame,

2) Rotating disc-bearer, driven by an anti-slip pulley,

3) Pendulum beam on which the pin is fastened, and its own axle, in line with the disc one,

4) Counter frame, which can be slided along the main frame columns up to the "contact" position,

5) Eccentric drum giving the β angle through which the pendulum rotates.

In order to determine where these differences came from, we repeated the experiments after different permutations on the rig parts.
For instance, keeping the main frame in position, we exchanged parts 2 (fig. 4) only, then parts 3, and so on..
Essentially these experiments have led to the following rig modifications:

-Change of the bearings of the pendulum axle: a small journal bearing was substituted by a special ball bearing,

-Rebalancing the eccentric drum (for the measurement by reluctance coils) at the 3 Hz oscillating speed,

-Increase of the accuracy of the cardan fastening system of the pin,

-Change in the precision of the installation of the counter frame (part 4) onto the main frame (part 1).

Although the general precision has been improved, allowing a good repeatibility of mean values of friction, a slight difference in the "graph behaviour" remains. That is: the "B" unit (right side on fig. 4) having a smaller pendulum damping (9 min. instead of 5.5 min.) tends nevertheless to flaten the friction signal (fig.5&5 bis). It is clear that, prior to reaching this conclusion, we checked that the electronics was not responsible.
We think that this difference comes from the difficulty to put perfectly in line the disc with the pendulum axle. At the present time the "B" unit has effectively a better alignment than "A". We are working on that problem.
Figure 5 gives an example of results:

-Disc in steel (0.87 C, 2 Mn, 0.3 Si, 0.15 V), four samples

-Pin in phosphorous bronze, four samples

-Load (α = 20°) 105 g/cm^2

-Same sequence as written above with 5 revolutions in place of 2 at the end of the sequence.

-Friction coefficient $/u = \frac{\sin /B}{\tan \alpha}$

It is noticeable that the "first two revolutions" give signals of the same aspect, there is a phase of conformity between the samples.

After the "running-in" at high speed, the "A" signal remains oscillating because the slider tends to describe a circle which intersects with the track on the disc engraved udring "running-in". This "radial interlocking" between pin and disc causes these oscillations. Besides this phenomenon, it is interesting to see how reproducible the friction coefficients are from one unit to the other. So it appears the "B" unit is more in line and the flat signal is true.

5.1.2. Intercomparison programme

A part of our activity has been devoted to the contracts, meetings and visits to the laboratories in order to prepare the technical aspect of an intercomparison programme on friction measurements. The test conditions and parameters were defined for the different apparatus whose geometries are:

-flat-on-flat (Leeds University)

-spherical-on-flat (B.A.M.)

-flat-on-cylindrical (T.N.O.)

The general experimental conditions are:

a) Materials

Because they are expected to have good dry-running properties, the following material conditions were chosen:

	Pin	Disc
1.	steel : SAE 52100	steel : SAE 52100
2.	steel : SAE 52100	bronze: Cu Sn 5 Pb 10
3.	bronze: Cu Sn 5 Pb 10	steel : SAE 52100
4.	carbon/graphite	steel : SAE 52100
5.	steel : SAE 52100	carbon/graphite
6.	carbon/graphite	bronze: Cu Sn 5 Pb 10

b) Number of experiments

Each laboratory will perform a total of 144 tests, repeating three times basic series of 48 experiments (6 combinations x 2 loads x 2 speeds x 2 surface finishes).

c) Experimental conditions

-Geometries	- line contacts (TNO) -spherical-on-flat (BAM) -flat-on-flat (Leeds)
-Friction track	-diameter 60 mm
-Environment	-synthetic wet air (70% h)

-Surface finishes (2)	-ground 0.2 /u CLA (P) -mirror polish 0.025 /u CLA . (0,2 /uP. T. V.)
-Cleaning	-ultrasonic at JRC Ispra, followed by 4 Soxhlet extractions in the laboratories (acetone) (not for graphite) (see 6.4)
-Loads (2)	-4 newtons (P) -40 newtons
-Speeds	-0.01 m/s (P) (duration: 28 h) -0.3 m/s (P) (duration: 1 h)
-Temperature	-room (23°C)
-Distance sliding	-1 km (in every case, even where a steady state is attained earlier)
-Nb of experiments	-144 ((12 + 36) + 48 + 48
-Measurements	-surface roughness (JRC-Ispra) -load -speed -friction force or coefficient -slider temperatures (in two positions) -humidity
-Recordings	-friction force (paper and magnetic tape) -vibrations -roughness (before and after test)

A preliminary 12-test series will be performed with the aim of producing friction-time records. A study of these records will enable the participants to decide upon the necessary experiment duration, as well as the desirability of tape-recording and computerized analysis. This will be related to the 4 N Load, 0.2 /um CLA roughness and to the two speeds (P).

5.1.3. Specimen preparation

The JRC Ispra, will provide each laboratory with the specimens necessary for the Phase One programme, including a surplus of 12.5% with respect to the basic quantity or quantities scheduled.

	DISCS			PINS		
	Br.	Gr.	Steel	Br.	Gr.	Steel
Basic	16	8	24	8	16	24
Basic x 3	48	24	12	24	48	12
(Basic x 3) x 1.125	54	27	18	27	54	18
Total :		162 Discs			162 Pins	

The JRC-Ispra, will provide the specimen in conformity with the respective drawings, "ready to use", except that the operator should clean them again at the precise time of testing, by means of a Soxhlet extraction with acetone (four times).
The carbon/graphite specimen will not be cleaned by acetone extraction. They will be allowed to reach equilibrium by exposure for 24 h to let the test atmosphere prior to the test.
In the case of spherical specimens for BAM, the pieces will be sent to Berlin to be shaped on a special apparatus.

5.1.4. Friction measurements in hostile environments

The apparatus Tr II as described in (2) has been used for friction measurements in air up to 200° and 400°. The experiments undertaken in the frame of a contract are now finished; they allowed the selection of materials having good friction properties in dry conditions, like polyacrylonitrile graphites.

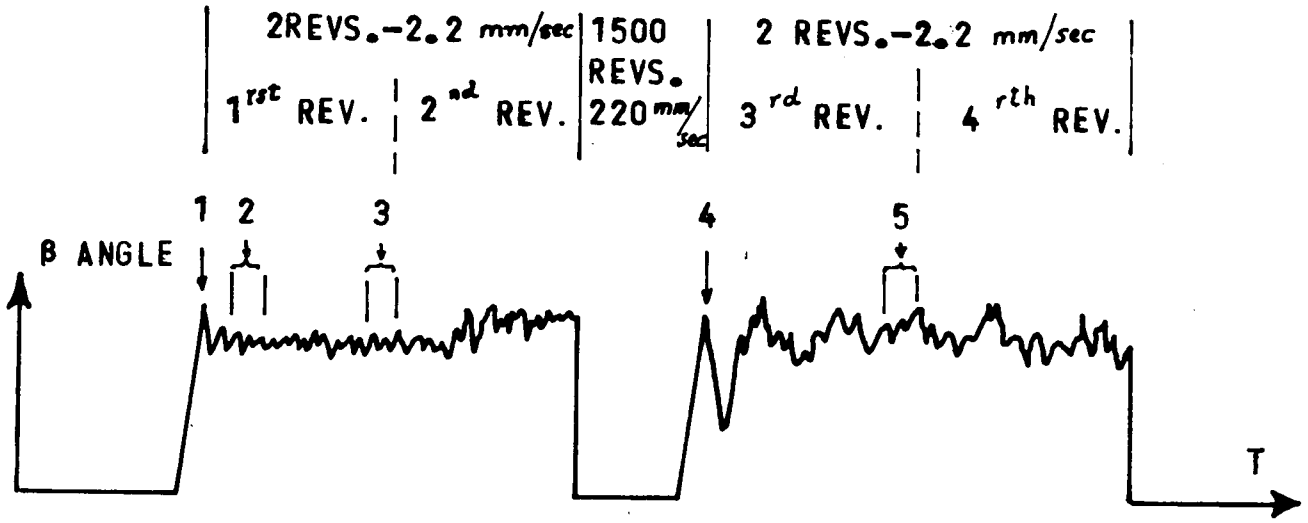
A special tribometer for measurements in water is in preparation and could be ready by February 1975.

The simulation experiments in sodium are beginning, the problem being to appreciate the wear on the lower, centering component of a fast reactor fuel assembly.

A special device has been projected and built to simulate the fretting-corrosion occurring between a fuel-pin and two grid-dimples in argon on gas breeder reactor.

5.1.5. References

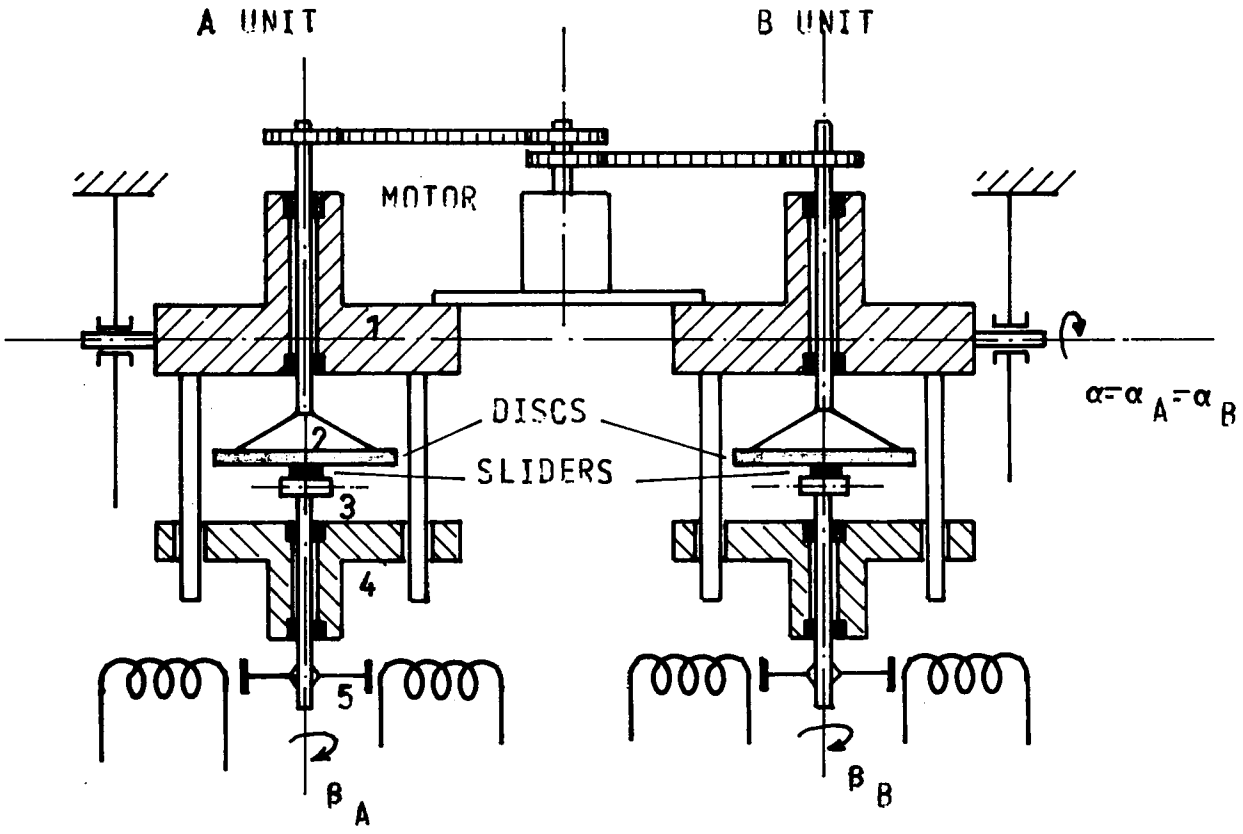
1) J.R.C. Annual Report 1972 page 267
2) "Tribology 1973 Activity report" B. C. d'Agraives, J. Toornvliet, Communication in press



1. FIRST PEAK CORRESPONDING TO THE STATIC FRICTION COEFFICIENT
2. EXTREME VALUES AFTER 10 ÷ 15 SEC. FROM 1
3. EXTREME VALUES DURING THE LAST 5 SEC. OF THE 1rst REVOLUTION
4. FIRST PEAK AFTER RUNNING-IN
5. EXTREME VALUES DURING THE LAST FIVE SECONDS OF THE 3 REVOLUTION

FIG. 3

BASIC SEQUENCE FOR DRY FRICTION MEASUREMENT.



1. MAIN RECLINING FRAME, BEARING THE DISC AXLE
2. ROTATING DISC-BEARER, DRIVEN BY ANTISLIP PULLEY
3. PENDULUM BEAM BEARING THE SLIDER AND ITS AXLE
4. COUNTER-FRAME MOUNTED BY MEANS OF 3 GUIDES ON THE MAIN FRAME
5. EXCENTRIC DRUM AND COILS FOR β MEASUREMENT

FIG, 1

SCHEMATIC VIEW, FROM ABOVE, OF THE 2 UNITS

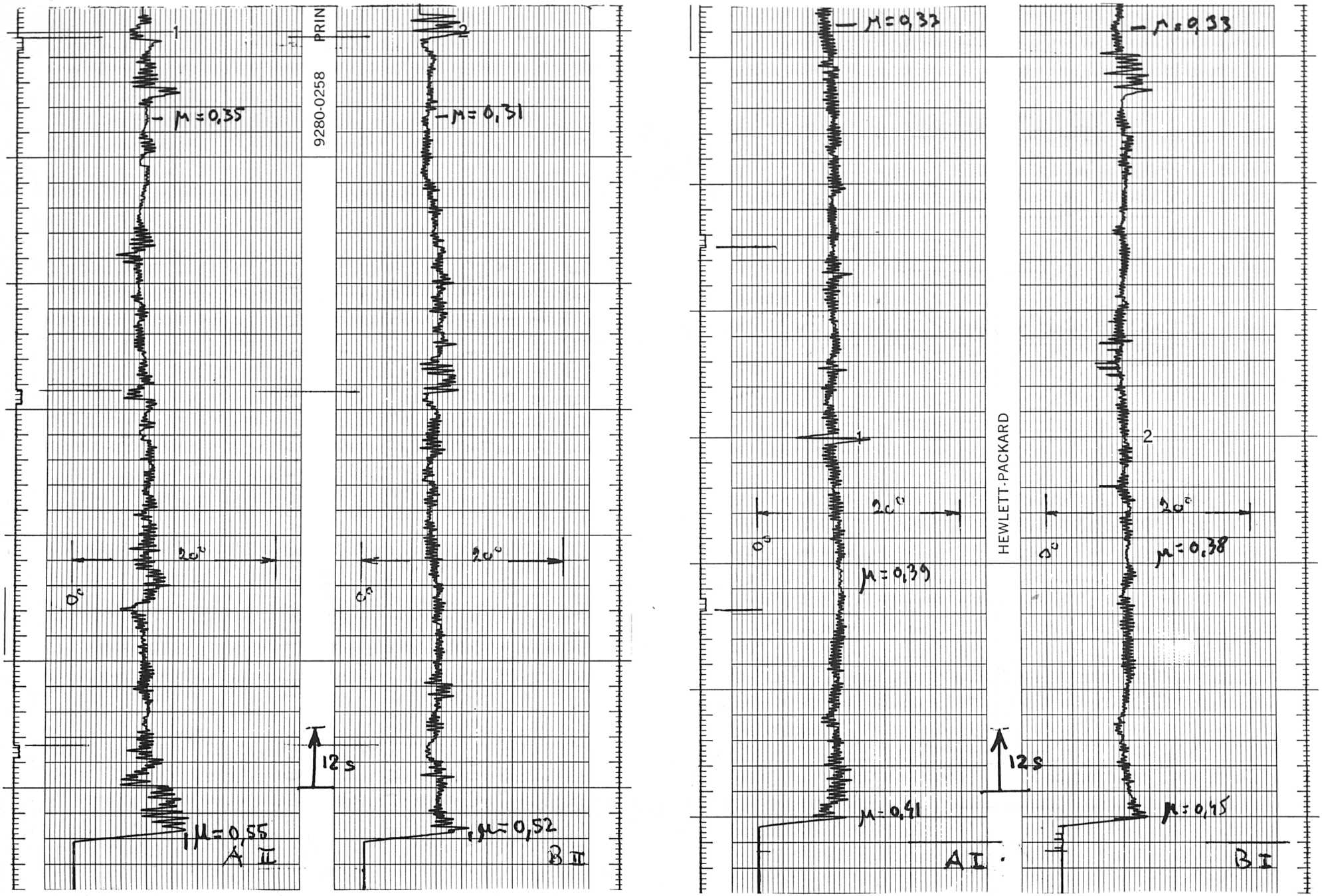
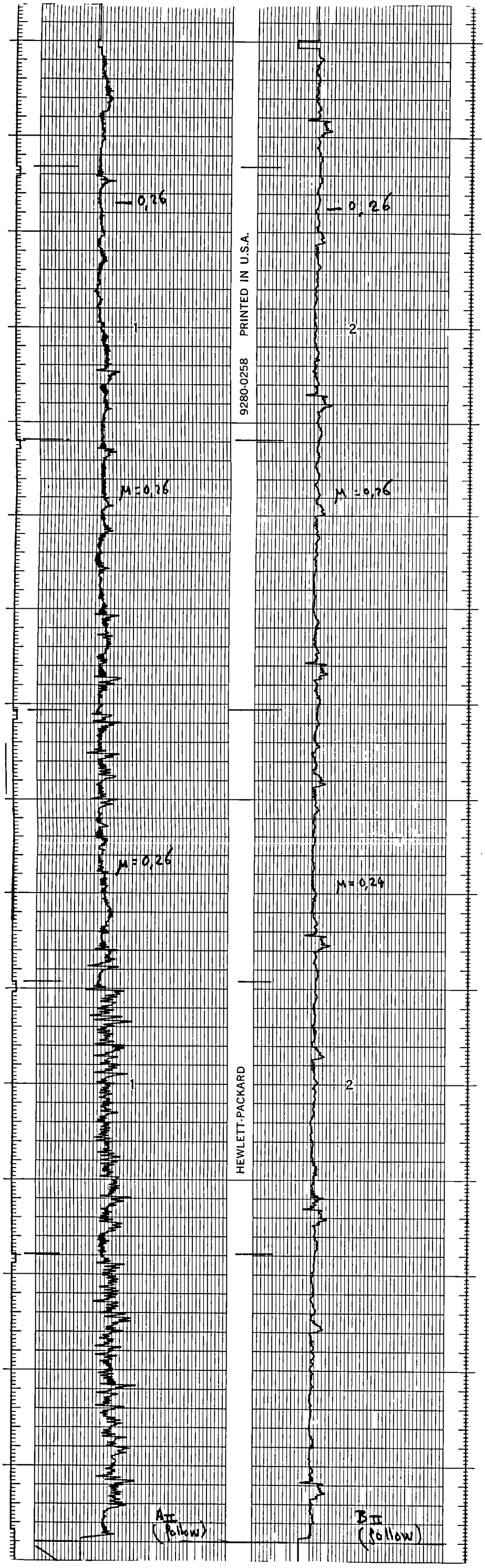


Fig. 5 (first 2 revolutions)



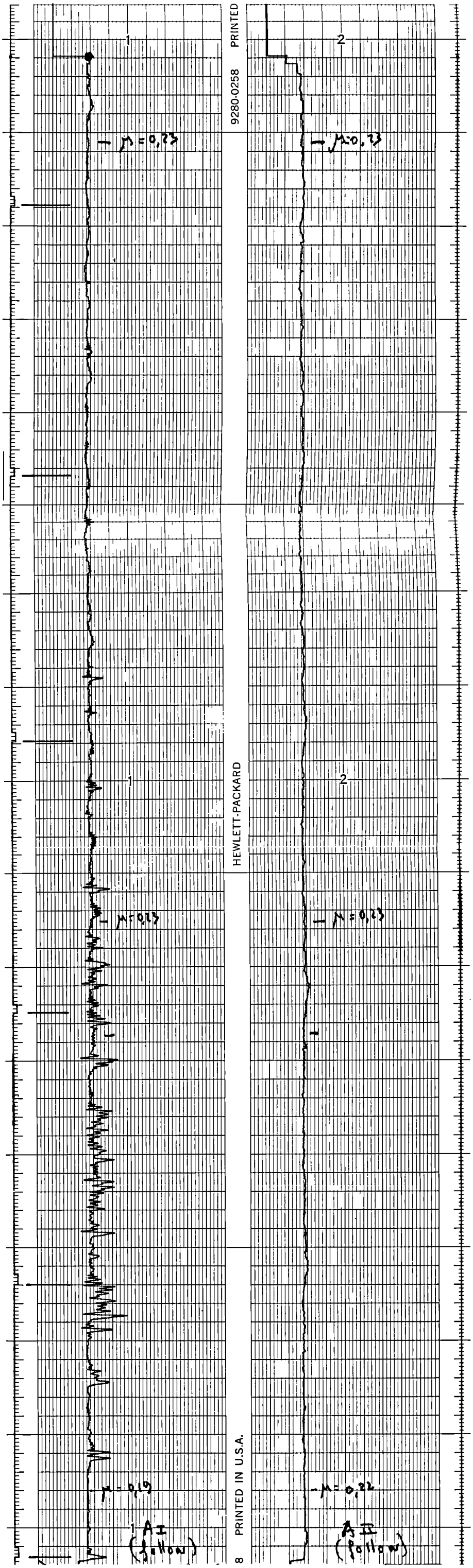


Fig. 5 bis (last 5 revolutions)

5.2. Reference microdefects and ultrasonic transducer characterisation (P. Jehenson, Borloo E., Bredael I.)

In the metallurgical industries the detection of defects by means of ultrasonics is carried out in a rather primitive manner. The lack of knowledge of characteristics of the transducers employed, (together with their relative electronic equipment), and the lack of reference samples leads to very unreliable measurement results. The aim of this study is therefore:

-to make available to the industry some well-defined reference defects,

-to characterise the transducers, as well as the electronic chains (equipment) associated with them.

5.2.1. Present state of our laboratory activities

a) Reference defects

Our work is particularly directed towards very small defects, since the production of large defects does not pose any special technological problems. For very small-diameter claddings, with thin walls, the punching technique developed in our laboratories gives complete satisfaction. In a cladding of about 6 mm diameter, and 0,4 mm wall thickness, internal and external defects of 1 mm long and 0,1 mm wide, and 20 and 40 µm depth can be produced, either longitudinally or transversally, and with excellent reproducibility.
A study is in progress to determine the ideal shape for such a defect, as a function of the ultrasonic reflectivity, depending on the detection techniques employed. This problem must be discussed by the existing BCR group of specialists, and could even become the object of a contract, in the context of indirect action. Alternative microdefect fabrications procedures are also being studied, and the most interesting results obtained so far are those involving a combination of electroerosion and ultrasonic machining techniques.
This technique is far more flexible than the punching method, and the geometry of the defects currently obtained have convinced us that this solution is very interesting.

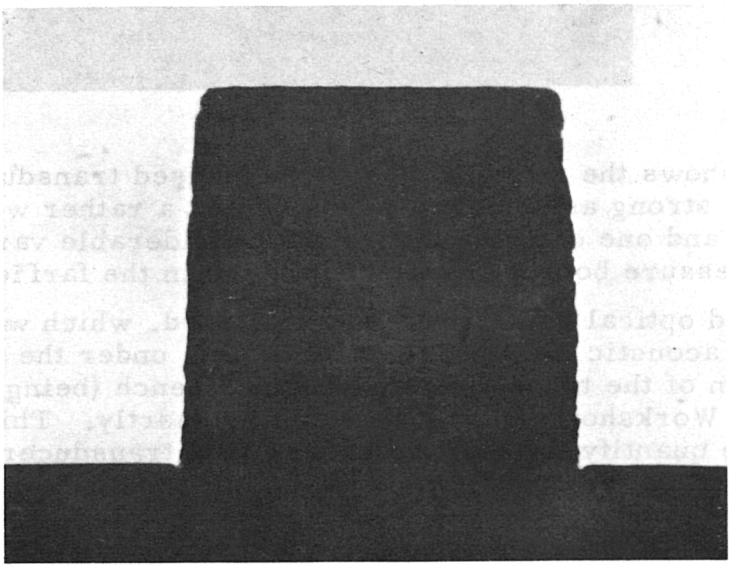


fig. 6

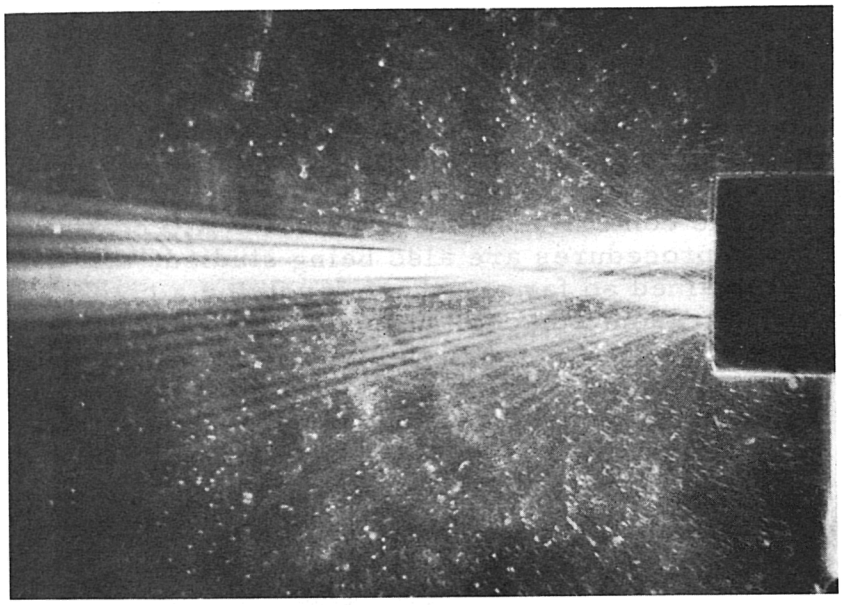
The fig. 6 represents an enlargement (50 x) of a cut of a cylindrical hole, with flat bottom of 1 mm diameter, and 1 mm depth. The reader should note the quality of this flat bottom, obtained after 5 rectifications of the cutting tool during the fabrication of this reference defect. The sharp angles obtained should also be noted.

This technique also offers the advantage of being easily applied to defects either on tubes or on flat pieces. The form of these defects can be changed simply by an appropriate choice of electrodes. As far as the verification of the geometrical quality, and the surfaces of the fabricated defects are concerned, a "replica" examination procedure (centrifugation) and extraction by compressed air is being carried out.

5. 2. 2. Characterisation of the transducers

Our laboratory equipment has enabled us to develop a technique using visualisation of the ultrasonic beam by the "schlieren" method. For this visualisation, the transducer under examination is excited by a continuous wave, which does not exactly simulate the true utilisation conditions, but nevertheless gives some useful data on the geometrical characteristics of the beam.

fig. 7



The fig. 7 shows the acoustic field of a focused transducer. One can observe the strong asymmetry of the beam, a rather weakly-defined focal zone, and one can clearly see the considerable variations in the acoustic pressure both in the nearf ield and in the farfield.

A second optical bench is being assembled, which will make the analysis of the acoustic field possible, this time under the true conditions of utilisation of the transducers. Another bench (being constructed in the Central Workshop), should be supplied shortly. This version should enable us to quantify the emitted energy by a transducer, and produce beam plots of any desired section of the ultrasonic beam.

The measurement of the electrical characteristics of the transducers continues, and the arrival of certain equipment ordered at the beginning of the year will enable us to make progress with this study.

5.2.3. Preparation of the indirect action

a) During this year the group of specialists met three times. Two new specialists joined the group, namely: one representative of the RTD (Netherlands) and one from the CEA (France). During the meetings this year, the directives on direct and indirect actions for 1974 and 1975 were discussed. A proposal for indirect action, proposed by the SERAM, was discussed and adopted. The final form of the questionnaire has been defined, and discussions on measurements of transducer characteristics are in progress. The next meeting is planned for February 1975.

b) Circulating measurements

Transducers measurements are continuing. Having been to Harwell and to the BAM, the transducers are at present in the hands of the RTD, Rotterdam. The laboratories of the CEA at Cadarache have agreed to take part in the round robin measurements on artificial defects. Once the Belgonucléaire have completed their measurements the reference defects will be sent to CEA Cadarache, where they will be characterised both by ultrasonics and by Eddy currents.

5.2.4. Publications and aims for 1975

- Report on the 2nd, 3rd and 4th meeting of the "Specialists on reference defects and transducer characterisation".

Continuation of the activities in progress, and widening of contacts. A contact is already envisaged in January 1975 with ASTM, and in March 1975, our BCR activities will be presented at Ultrasonics International 75. Further contacts with the Danes and the Italians.

5.3. Impact Toughness (D.J. Boerman, D. Quataert , G. Bouvelle)

The aim of the programme on impact toughness is to explore the feasibility of the development of a series of standard specimen for the testing and the monitoring of impact test machines in order to integrate and in some cases to replace indirect (metrological) methods. Several series of about 50 to 100 steel specimen of a given energy level will be broken under standardized conditions on machines of different design and origin within the European Community, but all satisfying the most stringent standards available. The I.S.O standards will be taken as a basis, since the european countries of the EEC have adhered to the I.S.O. recommendations on impact toughness. For technical reasons some prescriptions of the I.S.O. standards may be altered if judged necessary (for some dimensional tolerances of the anvils the more stringent ASTM tolerances will be preferred).

The first part of the programme will consist of the indirect testing of several machines (two machines will be tested and modified in CCR-Ispra, one in L.N.E.-Paris and one in B.A.M.-Berlin). In the meantime the specimen for the ring tests will be prepared by C.C.R.-Ispra (heat treatment and machining).

5.3.1. Summary of results

5.3.1.1. Verification of Wolpert II impact test machine

As described in our activity-report of 1973, we verified and modified the 12-years old Wolpert I tester (PW 30/15, machine n°609) existing in our laboratory. The principal axis and the fixation of the pendulum on this axis was in a very poor state of finishing with clearance between the axis and the pendulum. So we installed on a newly built foundation an identical Wolpert II tester (PW 30 30/15, machine 672), borrowed from another CCR-laboratory.

The metrological verification was made in the same way as described in our activity report of 1973. Three types of verifications caused important modifications.

The center of percussion: the oscillation period T = 1,77570 sec. for the original 15 mkg pendulum; which corresponds with a reduced length L_{red}= 782,47 mm, which falls out of the standardized tolerances. For the original 30 mkg pendulum we found T = 1,77883 sec., giving L_{red}=785,23 mm, which is out of standards too!

The energy-scale: with the cathetometer we verified the energy-scale and we found an error of 1,5 degrees in angle, caused by an eccentricity between the center of the scale and the center of the pendulum axis.

Parasitic energy losses: the free space between pendulum and the anvils is only $\frac{40 - 16}{2}$ = 12 mm for the broken specimen halfs (with a section of 10 by 10 mm and a diagonal = 14,1 mm) and we found out that this small space would cause parasitic energy losses.

Modifications for the 15 mkg pendulum: in principle the center of percussion was modified in the same way as described in our activity-report of 1973. This time the calculated corrections were:

$$m_1 = -270 \text{ gr}$$
$$m_2 = +1164,5 \text{ gr}$$

This resulted in an experimental value of $L_{red} = 798,68$ mm which is within standards.

In the original Wolpert-design, there are no tolerances and no mating parts foreseen in order to avoid the eccentricity of point 2.1.2.2. So a very careful mounting procedure was necessary.

We diminished the original thickness of the knife and of the pendulum-disc from 16 mm to 12 mm of the knife first and then a further thinning of the knife to 10 mm and a gradual decrease from 16 mm at the rear of the disc to 10 mm at the opposite side of the disc.

Experimental characteristics after modification

$L = 0,79973$ m
$d_1 = 0,70003$ m
$d_2 = 0,89996$ m
$P_1 = 9,581$ kp
$l_1 = 0,78247$ m
$P_2 = 10,317$ kp
$l_2 = 0,79868$ m

$h = 1,45391$ m
$\alpha = 144°53'07''$
$V_{impact} = 5,34$ m/sec

$E_{friction} = 0,04$ mkg
which is 0,3% of 15 mkg

all verifications as described in our activity report of 1973, points 4.1 till 4.14 (page 12 till 35) were repeated on the Wolpert II machine in 1974, confirming that the Wolpert II machine is at least as good within the standards as was the Wolpert I machine. (For significance of symbols see our activity report of 1973).

Direct verifications: the direct verification was made with the american standard specimen with warranted impact values, as described in our activity report of 1973. Just as in 1973 with the Wolpert I machine we are:
- out of limits for the low energy value
- within limits for the mean energy value
- within limits for the high energy value

These results are reproduced in the following table.

5.3.1.2. Verification of the new MFL impact tester

Both Wolpert impact testers were bought in 1962 and constructed according to the DIN-standards of 1952 and 1957. As explained in the introduction we need a machine satisfying the actual standards, so a new tester was ordered at Mohr-Federhaft-Losenhausen (MFL) in Germany by the end of 1973. Partly this machine should have closer tolerances than foreseen in the actual DIN-standards of 1973.

IMPACT TESTS AT - 40°C WITH CALIBRATED SPECIMEN.

Specimen		Measured total consumed energy		Corrected measured energy for friction losses=0,044 mkg		Warranted standard mean value E		Actual deviation ΔE	Notes
Series	N°	β	E	E	E	$E_{mean} / 2\sigma$	E_{mean}	permitted deviation ΔE from E_{mean}	Δ E	
		degrees	mkg	mkg	ft-lb	ft-lb	ft-lb	ft-lb	ft-lb	
J 11	635	124,2	2,112	2,068	14,957	14,310 / 1,988	12,8	± 1,00	+ 1,510	Out of limits
	663	125,0	2,017	1,973	14,271					
	669	125,9	1,912	1,868	13,508					
	689	123,5	2,202	2,158	15,606					
	693	126,0	1,870	1,826	13,210					
K 11	822	94,0	6,180	6,136	44,390	44,092 / 2,874	42,6	± 2,13	+ 1,492	within limits
	853	95,2	6,002	5,958	43,092					
	875	92,4	6,404	6,360	46,002					
	890	specimen not centered								
	987	95,4	5,973	5,929	42,885					
M 11	451	68,35	9,794	9,750	70,519	70,624 / 1,161	69,4	± 3,47	+ 1,224	within limits
	911	68,35	9,794	9,750	70,519					
	945	67,2	9,946	9,902	71,627					
	947	68,6	9,760	9,716	70,276					
	991	68,7	9,746	9,703	70,179					

A first provisional technical reception was made the 8 and 9 of July 1974 in the factory in the presence of Mr. Boerman and Mr. Bouvelle. We discovered an angle shift between the pendulum and the indicator, caused by a faulty fixation of the pendulum on the axis. A second and more complete verification began at Ispra in September and October of 1974, utilizing our own gauges, the cathetometer and other instrumentation. We discovered that the anvil distance was out of tolerances and that there exists an axial play of the pendulum greater than 0,4 mm (caused by the application of the electro-magnetic brake).

With the aid of an engineer of MFL modifications will be made here at Ispra in January 1975 in order to eliminate this failures.

5.3.2. Fabrication

5.3.2.1. Impact test machine

- Two new concrete foundations were made for the impact test machines,
- A special device was constructed for mounting the Wazau dynanometer, in view of a precise determination of the pendulum momentum,
- A device for the measurement of the length of the pendulum,
- A gauge for determining the distance between the anvils,
- A centering gauge for the knife edge of the pendulum,
- A cryostat made by Angelantoni in Milan has been adapted for the purpose of obtaining stabilized low temperature on six specimen holders.

5.3.2.2. Specimen

- 550 impact test specimen were milled, and heat treated in our laboratory. The final machining including the groove was done by the firm AZETA of Baveno,
- 3 sets of 5 gauges were made for the dimensional control of the specimen.

General remark

The main technical difficulty, which delayed to a large extent the beginning of the testing of impact specimen consisted in having a machine available satisfying the current I.S.O. and/or ASTM standards. Two existing machines did not completely satisfy the standards and a newly bought machine is currently modified in order to satisfy existing standards.

5.3.3. Publications related with the present programme

a) Standards

- ASTM E 23 - 72
- ISO R 148 of 1960 and R 442 of 1965
- DIN 50115 of 1970 and 51222 of 1973.

b) Publications

-N. H. Fahey
"The Charpy Impact Test. Its Accuracy and Factors affecting Test Results".
ASTM - STP n°466 of June 1969, page 76-92.

-D. E. Driscoll
"Reproducibility of the Charpy Impact Test"
ASTM-STP n° 176 of June 1955, page 70-75.

c) Working paper

Our activity - report of 1973 (Communication, in press).

ANNEX I

B.C.R. DATA BANK

by Maurandy C.
Bertolo C.

1. INTRODUCTION

Within the framework of the diffusion of information carried out by the Community Reference Bureau and following the publication of various catalogues on reference materials and high purity substances, the services of the JRC have undertaken, on behalf of the BCR Secretariat, to establish a data bank relating to these products.

This document outlines the position and the present state of work.

This bank should in the future form the basis for a Central Information Bureau for reference materials, which could later be linked up with the European tele-information network. *

The system foreseen would, amongst other things, permit:

-data storage,
-automatic retrieval of numerical or non-numerical data,
-automatic updating,
-conversational retrieval,
-automatic issue of information and the management of the operations performed by the bank.

After a study of the different systems available and the time factor having been taken into account, the choice fell on the SIMAS system, which is already in operation in the Centre for other data banks. This system will be used during the running-in period of the card-index, and although in its present form it does not exactly correspond to our requirements, it can be made more appropriate with some modifications.

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* A study of the technical problems arising from the creation of such a system and its connection with the already existing networks is being carried out under the supervision of the Commission. CETIS of Ispra is taking part in this study.

It is useless to describe the SIMAS system in detail since it can be found in other specialised papers.* We would simply point out that, for us, two essential transformations have already been carried out. The first permits the system to accept the numerical values and to apply them to a quantitative retrieval of any Boolean comparison expression($<$, $>$, =, etc.). The second permits the introduction of data on the inverted file, thus making possible the simple application of the same elementary information to numerous items. Other conversions which are important for the alphabetic retrieval of the items and for the automatic printing of the results are in course of being examined.

2. FORMATION OF THE DATA BANK

The bank is composed of two distinct card-indexes:

2.1. The items forming the bank itself are filed in the first card-index. They are divided into two different groups. The reference materials and the high purity substances are filed in the first group. The second groups is composed by the laboratories, companies and organisations that are interested in the materials of the first group or by the BCR activities. Therefore, those producing, selling or using reference materials, the laboratories contacted at the time of the enquiries, etc., are found in this group.

The card-index thus composed will permit a more flexible and easier management of the Secretariat activities, in addition to the more rapid diffusion of the information on reference materials.

In each of these groups, the information attributed to the items is of a different nature. There are four types of information, which can be classified as follows:

-the identifier(s) which identifies the item and which can only appear once in the card-index. This category, for example, covers the number recorded in the bank, in some cases, the name of the object, etc.

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* SIMAS, an interactive information system(International Computing Computing Symposium, Venice, April 1973)

A CETIS-SYNTAX report is about to be published

An alphanumerical retrieval can be made with this information.

-the classification which is codified information possessing several levels of hierarchy permitting a ramified indexing of the items.

-codified information by means of key-words and corresponding thesauri. The numerical information is to be found in this category. The corresponding numerical value and the unit used are associated with key-word indicating the component (or the property).

-lastly, the literal information which, contrary to the above, is not used for the automatic retrieval of the items but as addressing or complementary information supplied to the requester.

2.2. A second card-index associated with the first includes the laboratories, firms and organisations using the data bank. Each customer may, after obtaining the necessary authorisation, consult any part of the card-index directly; this facility will, in the future, be used for teleprocessing. The questions are recorded by the system which supplies a detailed analysis of the statistic statement of the questions.

The information is reduced to a minimum in this card-index:

-the name for the alphabetical retrieval,
-the authorisations (type of retrieval permitted, authorised groups and classifications),
-the address,
-the name of the correspondent,
-the country.

We can now examine in more detail, the information supplied for the two groups of the first card-index. It should first of all be mentioned that the information given in the following descriptions does not always have its full meaning for all types of items. Some information, for example, which is essential for identifying a chemical product, can be without meaning or can have a more or less different significance when applied to a technological reference material. This is to say that by billing the cards of each item, only the significant parameters are taken into consideration.

3. REFERENCE MATERIALS AND HIGH PURITY SUBSTANCES GROUP

The information attributed to the items of this group is detailed below. Within a given group of information (codified for example) the items of information are independent of each other, and placed in any order.

3.1. The identifiers

3.1.1. BCR identification number: this is an increasing number starting from 1, given to each item when it is recorded by the bank.

3.1.2. The name(s) of the item: this information will be used especially for the organic chemical products, the pharmaceutical products and those to be used in the alphabetical retrieval for the items. There is the possibility of adding a second name for products with different designations.

3.2. Classifications

These are in effect codified data or key-words forming a 3-level hierarchy. This possibility offered by SIMAS was used to classify the products into groups, sub-groups, etc. according to their nature. An example of this classification is given in the appendix.

3.3. Codified information

All the following codified data can be used in the automatic retrieval of the items. The choice is such that the data bank can reply to most of the reasonable questions addressed to it by the users of the bank. An example of some typical questions is given in the appendix.

3.3.1. Name of producer: all the names of the firm, the products of which are recorded in the data bank, are codified. The corresponding thesaurus should contain some 300 key-words.

3.3.2. Methods: the type of method, measurement/analysis (or the particular method) for which the material will be used, are indicated.

3.3.3. State/phase/form: the physical state of the sample is given in a codified manner as well as its form (solid), its crystalline state, etc.

3.3.4. Chemical formula: this is a simple way of finding the chemical products (especially inorganic) which appear in the card-index. If a simple substance is involved, the code name of the corresponding element is given. If a molecule is entailed, the code names

of all the component elements are indicated, each followed by a number representing the number of atoms of the element in question in the molecule. For example, to find a sulphuric acid sample, samples having the following characteristics are looked for:

sulphur (1) oxygen (4) hydrogen (2)

This imperfect but simple procedure covers satisfactorily almost all the inorganic chemical products listed in the card-index for the time being.

At present, an extension of this procedure to include organic chemical products is being examined.

3.3.5. Purity: this factor is used to express (in %) the purity of certain simple or compound chemical substances.

3.3.6. Properties: for each certified property a group of 3 items is given, that is:

-the code of the certified property
-the certified numerical value
-the unit used

Providing that the request is made in a coherent unit, the system itself effects the unit conversion for the retrieval and retranslates it into the calling unit to give the results.

3.3.7. Composition: as in the preceding paragraph, groups of 3 items are given, that is:

-the code of the component (principal or impurity)
-the numeric value of the contents
-the unit used (%, ppm, etc.)

In this paragraph, the groups of data can be as many as thirty (in the case of spectrographic metallic samples).

3.3.8. Certification: a certain number of indications relating to the certification process followed by the producer is given to enable the requester to have some idea of the certification of the sample. Care must be taken not to identify this "idea" being a velue judgement since there are too many factors that are not taken into consideration: measurement quality, laboratory experience and specialisation, etc.

3.3.9. Availability: this parameter indicates whether the sample in question is in stock, on order, if the stock is being renewed, if the production has been abandoned, or if another sample replaces the one concerned.

3.4. Literal information

In this group, information is found which is of no use for retrieval whether it be a very particular information (scientific or technical article concerning the material identification number of the producer) or information which is of no interest to the retrieval (for example, the price).

The information covered by this group is:

3.4.1. Description: this is a very brief description of the material using as far as possible the terms (or their exact translation) employed by the producer.

3.4.2. Identification number of the producer

3.4.3. Bibliography: all the useful bibliographic information is supplied to the requester relating to the material requested and to the work performed during its study and development: articles, reports, etc.

3.4.4. Standards: the standard reference with which the item has a direct relationship is given (when, for example, the item is specially developed to control or satisfy a certain standard).

3.4.5. Conditions of use (if any): the possible limitations in the use of the sample are indicated: temperatures, pressures, chemical environment, etc.

3.4.6. Weight, volume, dimensions: the weight, dimensions and/or volume of the sample are given. It has been seen above that the state, phase or form (solid) of the sample is given in a codified form in order to enable the retrieval to be made for samples having a given form.

3.4.7. Estimated price: the price quoted by the producer at the last updating of the cards is given. Even if a not very recent date is involved, the connection of these two data gives an idea of the actual price.

3.4.8. Updating: date of the last updating of the card. In principle, all the information is revised at this date.

4. ASSOCIATED LABORATORIES AND ORGANISATIONS GROUP

As previously mentioned, this group covers all the laboratories, firms and organisations concerned in the reference materials and the ultra-pure substances on one part and by the BCR activities on the other.

Amongst other things, when a material has been found in the banl that complies with the requirements, it can supply all the references of the producer and the different agents in the Community countries. It also facilitates the management of the activities of the Secretariat, in particular, during the enquiries, distribution of circular or brochures, etc.

The following main groups of information can be found on a typical card of this group:

4.1. The identifier (name)

This is the name of the company or laboratory written in a form which places in evidence the most significant words (paper names, name of specialities, etc.). The name, (in the order in which it is written on the card) is used in the alphabetic retrieval. If the head office of a firm is involved, only the name is written. If a laboratory, a department, etc. (obviously not having the same address as the head office) a number follows the name (the same as that of the head office), increasing from 1 to N, as the cards meet.

4.2. The classifications

This is the same as that encountered in the materials group. The same groups and sub-groups of products are found. In this case, however, it deals with the products of interest to the laboratories. This information will be used to automatically address brochures and pamphlets.

4.3. Codified information

This covers the information needed to retrieve firms conforming to certain criteria. A list of typical questions to which the card-index can quickly reply, whether in batch mode or in teleprocessing (teleprinter or video) is given in the appendix.

4.3.1. Activities within the field of the reference materials:
whether a producer, retailer, user, producer for his personal use, etc. is concerned is given.

4.3.2. Economic status:
in this case is indicated whether it concerns an official organisation, a private company (main office or subsidiary), a professional association, etc.

4.3.3. Industrial activity sector

4.3.4. Turnover

4.3.5. Number of employees

4.3.6. European classification:
These three latter items are supplied (when known) in order to estimate, in case of need, the weight that a demand may have (MR) or a reply (to an enquiry). Naturally this type of reasoning has to be done with great care.

4.3.7. Parent companies and associates:
when a subsidiary is concerned, the key-word(s) of the parent company(ies) is supplied.

4.3.8. Agent:
when the card refers to an agent, the codes of all the producers represented are given (of which, obviously the products appear in the materials card-index).

4.3.9. Documentation:
this covers, in a codified form, the type of circular sent to a firm, the replies and documentation received, or to know whether its products appear on the card-index, etc.

4.3.10. Enquiries:
for each enquiry made by the BCR Secretariat, this indicates whether the firm has been contacted, by questionnaire or by direct contact, whether it has answered, positively or otherwise, whether it is ready to collaborate, etc.

4.3.11. Distribution:
this section follows all the distributions of documents (reports, catalogues) performed at the laboratories and companies concerned. Later on it can be used to control the distribution of periodicals, brochures, bulletins, etc.

4.3.12. Subscription:
this paragraph can be used later to make possible subscriptions.

4.3.13. Country

4.4. Literal information

This group of information covers:

4.4.1. Postal address: this concernes the complete postal address written normally and which is exactly transcribed on the label to send off the brochures and correspondence.

4.4.2. Name of a first correspondent

4.4.3. Name of a second correspondent

4.4.4. Name of a third correspondent

This covers the names or persons to whom correspondence is sent and which are later added successively to the name of the company on the labels.

4.4.5. Telephone number

4.4.6. Telex number

4.4.7. Date of the last updating

All the literal information is passive information which only appears when the corresponding item has been extracted from the card-index.

5. OPERATION OF THE BANK

The bank can be used direct in batch processing whether with teleprocessing by means of a teleprinter or with a video screen.

The first mode of operation corresponds to the use made by the Secretariat when publications are issued or for replies to grouped questions.

The second mode is that which permits instantaneous replies to be made to an isolated question. It is obviously the one which will be used by clients linked to the future European teleinformation network. For the present, there are several versions of the teleprocessing utilisation programme. The first, the oldest, develops at the same time as the conversation between the requester and the system. The system explains each time the keys of the different codes and the possible successive questions.

The advantage is that even a person not very familiar with the bank may ask questions. The drawback is that this conversation takes a long time, especially when working with a type 2751 terminal.

A second version now being developed enables the questions to be made direct to the bank and the system supplies the reply immediately. The process is very rapid, but the requester must know thoroughly in advance all the codes of the system.

The search for items in the bank can be made either by the name (s) or by the Boolean expression (relationship between logical sums and products) of different parameters which characterize the items of a certain group.

To make things clearer, some typical questions that could be made to the bank during normal operation are given below.

6. TYPICAL QUESTIONS TO BE MADE TO THE DATA BANK

Note

The following questions are some typical examples demonstrating the structure of the questions that can be put. There is no need to look for the chemical, physical or technical significance of these examples.

6.1. Reference Materials and High Purity Substances Group

To be retrieved in the bank, when the materials corresponding to the following specifications appear:

- viscosity reference materials certified in the field 10,000/100,000 cP.
- samples of aluminium alloy complying with the following conditions:
  $0.5 < Mg < 3.00 \quad Cu < 0.08 \quad Si \leq 0.005$ (in %)
- samples of nickel having a purity of $\geq$ 99.95% supplied in the form of wire
- monocrystals of bismuth, cadmium sulphur
- samples of sulphuric acid accompanied by an analysis certificate (lot) and satisfying the following conditions:

$$\begin{aligned} As &< 0.001 \\ Cl &\leq 0.05 \end{aligned} \quad \text{(in \%)}$$

-samples of benzene having a purity $\geq$ 99.95%
-samples of argon having a purity of $>$ 99.95% and containing less than 5 ppm in volume of nitrogen
-solutions of ^{63}Ni
-sources of americium 241 having an intensity less than 1 millicurie
- γ-sources, γ-radiation $>$ 1.5 MeV
-samples of minerals having the following partial composition:
(in %) $45 \leq SiO_2 \leq 50$ $Al_2O_3 \geq 20$ $MgO < 5$
-methane+hydrogen mixtures containing less than 1% of hydrogen
-samples of Rockwell hardness, hardness included between 50 and 100 Rockwell units
-samples of thermal conductivity $>$ 0.3 W/m. °C
-organo-metallic composition having a certified percentage of silver
-sample of steel having a certified content of oxygen included between 50 and 100 ppm
-viscosity reference materials produced by company X
-organo-metallic type reference materials produced in the Community
-list of spectrographic stainless steel standard elaborated in the Community. These are classified according to the producer, and the producers according to the country
-list of samples of cobalt having a purity of 99.9%. These are grouped in the alphabetical order of the producers and for each producer according to the decreasing purity
-list of all the reference materials having certain physical properties

These are presented following the internal classification of the bank. In any one category, the products are groupes according to the producer, and the producers are in alphabetical order.

6.2. Associated Laboratories Group

To be retrieved in the bank, providing that they have been entered and supplied with the address, telephone number, name of the correspondents, laboratories, companies or organisations which satisfy the following conditions:

-those, the names of which are.....

-those representing in .....(country) the firm.....
-those to whom the report was sent.....
-those who offered to collaborate when the enquiry on....was made
-metallurgical companies manufacturing their own reference materials
-those interested in the reference materials within the nuclear field
-the research institutes interested in reference materials having optical properties

Note : once the retrieval has been made, the system automatically supplies on labels the list of names and addresses of the persons and companies retrieved.

6.3. Questions to the Users Card-Index

-how many questions were made to the bank by.....of.....at..... ?
-how many of the questions concerning viscosity reference materials were made in..... ?
-proportion of questions for composition reference materials compared with those for materials having physical or technological properties ?
-was the material.....subject of a question on such and such date ? How many times and by whom ?
-which are the customers having made more than .....questions in the past year ?

7. PRESENT STATE OF THE WORK AND LIMITS OF THE SYSTEM

At present, replies to all the questions made in the typical questionnaire cannot be obtained with the SIMAS system.

As already seen, a preliminary modification covered by a contract with SYNTAX has been carried out to enable numerical information to be introduced into the bank and a quantitative retrieval to be made. A table of unit (simple or compound) equivalences permits the input or output of information to be made in no matter what unit system considered in the table.

Another modification, which has formed the subject of a supplementary contract, enables groups of items linked together to be created.

In particular, this permits the attribution of the same patronymic name to a certain group of items and greatly facilitate the retrieval for products especially the organic chemical ones, the pharmaceutical products and those used for medical analysis.

Some printing sub-routines will remain to be written concerning the issue of catalogues, brochures, etc. This relatively less important work should be undertaken by CETIS.

However, this system does have a number of drawbacks for a data bank such as ours, since it should later on be open to the public, that is to say, access should be easy.

The main criticisms to be made to the system are:

-in the present system only one group can be questioned at a time: if it is desired, for example, to retrieve a particular sample and the references of its producer, this has to be done in two operations. In teleprocessing this is of little importance but in batch mode it requires two successive operations of the machine.

-we have seen that this system admits numerical values associated to a parameter (material properties, component element, etc.) but several values cannot be linked up with each other. We cannot, for example, associate a vapour pressure to a temperature or a refraction index to a wave length. Not being able to associate two values, we cannot define a range of values. In some cases this is a considerable drawback in the exact definition of the use of a material.

-since most of the information is in a codified form and the corresponding key-words permit the most varied items to be clearly defined, voluminous thesauri are used. It becomes tedious, therefore, both in input as well in output, to search for key-words to be used in the retrieval and the operation has to be performed by specialised personnel who know the system perfectly.

-the data input requires the manipulation of an excessive amount of cards. Roughly 50 cards are necessary to describe an item. For 8.000 samples + 2.000 associated laboratories, 500.000 cards are involved.

For these reasons we have already to think about the eventual substitution of the SIMAS by another system fitting better our needs, specially as far as the disponibility of the data-bank to non-specialized people is concerned. We are now looking for such systems actually in operation or in progress.

	CODE
CLASS - CLASS OF REFERENCE MATERIALS AND HIGH PURITY SUBSTANCES	AAQY
	CODE
FIELD - METALLURGICAL MATERIALS	AABI
1.SECTOR -	
FERROUS MATERIALS	AABJ
LIST OF CATEGORIES -	
PURE COMMERCIAL IRONS	AABK
UNALLOYED STEELS	AABL
LOW-ALLOY STEELS	AABM
HIGH-ALLOY STEELS	AABN
SPECIAL STEELS	AABO
CAST IRONS	AABP
FERRO-ALLOYS	AABQ
2.SECTOR -	
NON-FERROUS METALS AND ALLOYS	AABR
LIST OF CATEGORIES -	
ALUMINUM AND AL-BASE ALLOYS	AABS
MAGNESIUM AND MG-BASE ALLOYS	AABT
REFRACTORY METALS AND ALLOYS	AABU
PRECIOUS METALS AND ALLOYS	AABV
RARE EARTHS AND ALLOYS	AABW
MISCELLANEOUS NON-FERROUS METALS AND ALLOYS	AABX
COBALT AND CO-BASE ALLOYS	AA€U
NICKEL AND NI-BASE ALLOYS	AA€V
COPPER AND CU-BASE ALLOYS	AA€W
LEAD AND PB-BASE ALLOYS	AA€X
TIN AND SN-BASE ALLOYS	AA€Y
ZINC AND ZN-BASE ALLOYS	AA€Z
TITANIUM AND TI-BASE ALLOYS	AA€0
SELENIUM AND SE-BASE ALLOYS	AA€1
ZIRCONIUM AND ZR-BASE ALLOYS	AA€2
ACTINIDE SERIES METALS AND ALLOYS	ABAT
3.SECTOR -	
METAL SAMPLES FOR GAS MEASUREMENT	AABY
LIST OF CATEGORIES -	
4.SECTOR -	
ORES,CONCENTRATES,AGGLOMERATES	AABZ
LIST OF CATEGORIES -	
5.SECTOR -	
METALLURGICAL BY-PRODUCTS SLAGS	AABO
LIST OF CATEGORIES -	

	CODE
FIELD - INORGANIC CHEMICALS	AAB1
1.SECTOR - ELEMENTS LIST OF CATEGORIES -	AAB2
2.SECTOR - INORGANIC CHEMICALS IN GENERAL	AAB3
LIST OF CATEGORIES -	
ACIDS	AAB4
BASES	AAB5
AMPHOTERS	AAB6
OXIDES,HYDROXIDES	AAB7
OXYGENATED SALTS	AAB8
NON-OXYGENATED SALTS	AAB9
MISCELLANEOUS INORGANIC CHEMICALS	AACA
3.SECTOR - INORGANIC CHEMICALS OF SPECIAL APPLICATION	AACB
LIST OF CATEGORIES -	
FERTILIZERS	AACC
CEMENTS	AACD
GLASSES	AACE
REFRACTORIES AND CERAMICS	AACF
MINERALS	AACG
MISCELLANEOUS INORGANIC CHEMICALS OF SPECIAL APPLICATION	AACH
4.SECTOR - NUCLEAR MATERIALS	AACI
LIST OF CATEGORIES -	
NUCLEAR FUELS	AACJ
MODERATORS	AACK
ABSORBERS	AACL
RADIOACTIVE SOLUTIONS,RADIONUCLIDES	AACM
MISCELLANEOUS NUCLEAR MATERIALS	AACN

	CODE
FIELD - ORGANIC CHEMICALS	AACO
1.SECTOR -	
ORGANIC CHEMICALS OF GENERAL APPLICATION	AACP
LIST OF CATEGORIES -	
HYDROCARBONS	AACQ
HALOGENATED HYDROCARBONS	AACR
ALCOOLS,PHENOLS AND OXYGENATED COMPOUNDS	AACS
ORGANIC ACIDS	AACT
LIPIDS	AACU
CARBOHYDRATES	AACV
NITROGEN COMPOUNDS	AACW
SULPHUR COMPOUNDS	AAON
ORGANO-METALLIC COMPOUNDS	AACX
AMINO ACIDS AND PROTEINS	AACY
MISCELLANEOUS ORGANIC CHEMICALS OF GENERAL APPLICATION	AACZ
2.SECTOR -	
ORGANIC CHEMICALS OF SPECIAL APPLICATION	AACO
LIST OF CATEGORIES -	
PESTICIDES	AAC1
PHARMACEUTICALS	AAC2
FOODSTUFF ADDITIVES	AAC3
POLYMERS AND PLASTICS	AAC4
MISCELLANEOUS ORGANIC CHEMICALS OF SPECIAL APPLICATION	AAC5

	CODE
FIELD - REFERENCE MATERIALS FOR CLINICAL ANALYSIS	AAC6

	CODE
FIELD - PHYSICAL PROPERTIES	AAC7
1.SECTOR - PHYSICO-CHEMICAL PROPERTIES	AAC8
LIST OF CATEGORIES -	AAC9
DENSITY	AADA
VISCOSITY	AADB
VAPOUR PRESSURE	AADC
MOLECULAR WEIGHT	AADD
CORROSION	AADE
ION ACTIVITY STANDARDS PH,PD	AADF
MISCELLANEOUS PHYSICO-CHEMICAL PROPERTIES	AADG
2.SECTOR - ELECTRICAL AND MAGNETIC PROPERTIES	AADH
LIST OF CATEGORIES -	AADI
DIELECTRIC CONSTANT	AADJ
ANGLE OF LOSSES	AADK
MAGNETIC SUSCEPTIBILITY	AADL
ELECTRICAL CONDUCTIVITY	AADM
3.SECTOR - THERMAL PROPERTIES	AADN
LIST OF CATEGORIES -	AADO
TEMPERATURE FIXED POINTS	AADP
HEAT OF COMBUSTION	AADQ
THERMAL COMDUCTIBILITY AND RESISTIVITY	AADR
THERMAL RADIATION	AADS
ENTHALPY, SPECIFIC HEAT	AADT
INFLAMMABILITY	AADU
LATENT HEATS	AADV
4.SECTOR - OPTICAL PROPERTIES	AADW
LIST OF CATEGORIES -	AADX
ABSORPTION, TRANSMISSION	AADY
REFRACTIVE INDEX	AADZ
POLARIMETRY	AAD0
REFERENCES FOR COLOR/FREQUENCIES MEASUREMENTS	AAD1
LUMINESCENCE	AAD2
5.SECTOR - NUCLEAR PROPERTIES	AAD3
LIST OF CATEGORIES -	AAD4
ALPHA-EMITTERS	AAD5
BETA-EMITTERS	AAD6
GAMMA-EMITTERS	AAD7
ELECTRON CAPTURE	AAD8
NEUTRONS SOURCES	AAD9
SCINTILLATION COUNTING	
MOSSBAUER EFFECT	
MISCELLANEOUS NUCLEAR PROPERTIES	
6.SECTOR - MISCELLANEOUS PHYSICAL PROPERTIES	
LIST OF CATEGORIES -	
REFERENCE FUELS	

	CODE
FIELD - TECHNOLOGICAL PROPERTIES	AAEA
1.SECTOR -	
MECHANICAL PROPERTIES	AAEB
LIST OF CATEGORIES -	
HARDNESS	AAEC
TRACTION,COMPRESSION	AAED
TRIBOLOGY, WEAR	AAEE
DILATOMETRY	AAEF
TOUGHNESS	AAEG
MISCELLANEOUS MECHANICAL PROPERTIES	AAEH
2.SECTOR -	
CALIBRATION SAMPLES	AAEI
LIST OF CATEGORIES -	
THIN COATINGS	AAEJ
GRANULOMETRY	AAEK
TURBIDIMETRY	AAEL
STATE OF SURFACES	AAEM
RUBBERS	AAEN
SPECIFIC SURFACES	AAEO
PERMEATION TUBES	AAEP
GEOMETRICAL GAUGES	AAEQ
MISCELLANEOUS CALIBRATION SAMPLES	AAER

PUBLICATION BI-MENSUELLE

solution 1

Travaux effectués par le secrétariat BCR
agent cat. A ou B ..... 183,5 %
" cat. C ..... 25 %

Collaboration des secrétariats des groupes de travail BCR
% par secrétariat agent cat. A ..... 5 %

TRAVAUX DE COORDINATION — 25%

phase I PREPARATION

- recherche et sélection des articles — 12% — 3%
- photocopie des titres et résumés existant : 80 % — 5,5% 5% — traductions 15 % — résumés 5 % — 1%
- élaboration du bordereau — 2% 5%
- signalisation et indexation — 10%
- facultatif : commande et classement microfilms — 2% 5% — 1%

phase II COMPOSITION

- 10%
- 5% — enregistrement / contrôle enregistrement
- composition
- montage
- 2%

phase III INDEX AUTOMATIQUE KWIC

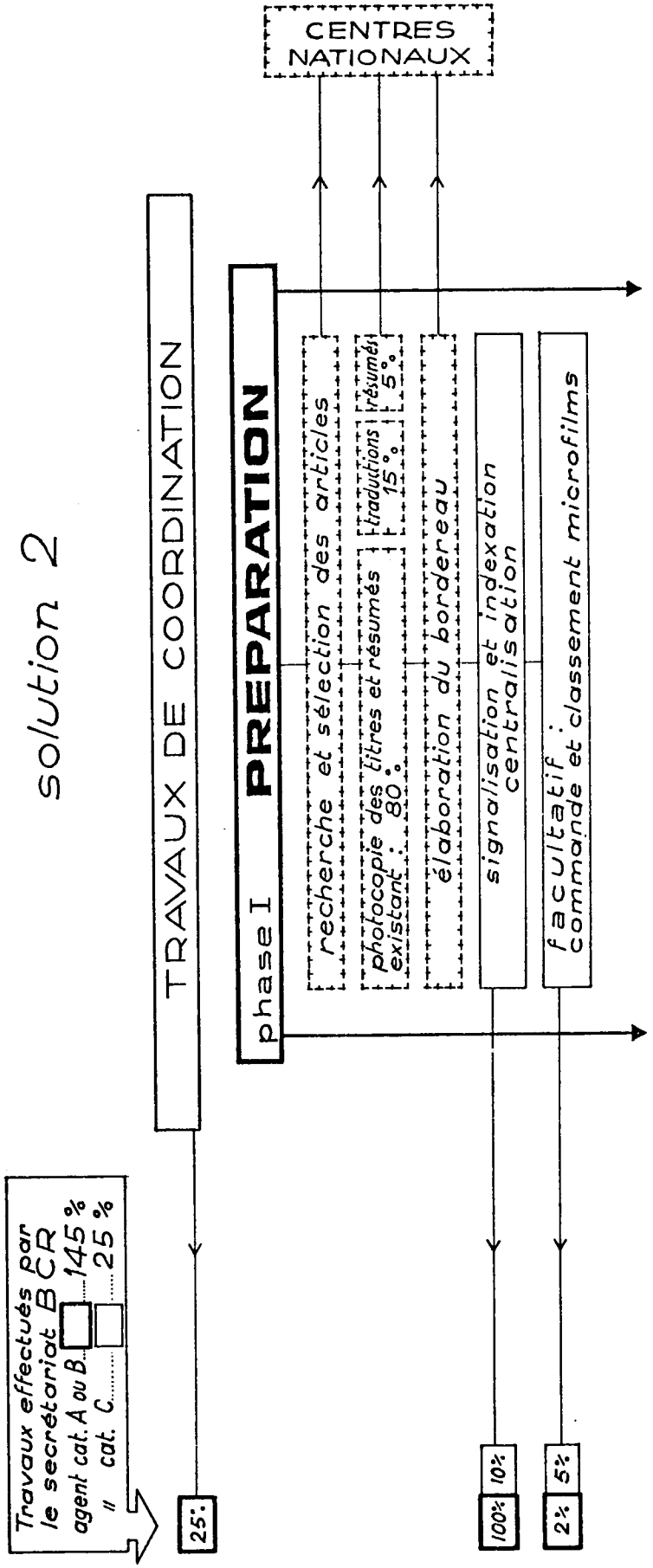
- analyse et préparation des données
- perforation
- passage en machine 1'
- analyse des résultats

phase IV PUBLICATION OFFSET DU BULLETIN

phase V EXPEDITION

- 1% — tenue à jour des adresses
- adresses autocollantes fabriquées par le CETIS
- 5% — collage et mise sous enveloppe pour 1000 adresses
- 5% — courrier secrétariat

ANNEXE II BIS



ANNEX III

I - PERIODICALS

ACTA CHEMICA SCANDINAVICA
A.I.Ch.E. JOURNAL
AMERICAN CERAMIC SOCIETY BULLETIN
ANALUSIS
ANALYST
ANALYTICA CHEMICA ACTA
ANALYTICAL BIOCHEMISTRY
ANALYTICAL CHEMISTRY
APPLIED OPTICS
APPLIED SPECTROSCOPY
ARCHIV FUER EISENHUETTENWESEN
A.S.T.M. STANDARDIZATION NEWS
ATMOSPHERIC ENVIRONMENT
BERICHTE DER DEUTSCHEN KERAMISCHEN GESELLSCHAFT
BRITISH JOURNAL OF NONDESTRUCTIVE TESTING
BULLETIN DE LA SOCIETE CHIMIQUE DE FRANCE
BULLETIN MENSUEL DE LA NORMALISATION FRANCAISE
CHEMICAL COMMUNICATIONS
CHEMICAL ENGINEERING
CHEMICAL ENGINEERING PROGRESS
CHEMICO-BIOLOGICAL INTERACTIONS
CHEMISTRY IN BRITAIN
CHIMICA E L'INDUSTRIA
CLINICA CHIMICA ACTA
COURRIER DE LA NORMALISATION
FORTSCHRITTE DER PHYSIK
FRESENIUS ZEITSCHRIFT FUER ANALYTISCHE CHEMIE
GAZZETTA CHIMICA ITALIANA
HELVETICA CHIMICA ACTA
INDUSTRIAL LABORATORY (URSS)
INTERNATIONAL JOURNAL OF ENVIRONMENTAL ANALYTICAL C
INTERNATIONAL LABORATORY
INTERNATIONAL METALLURGICAL REVIEWS(THE INSTITUTE OF
JOURNAL OF BIOLOGICAL CHEMISTRY

JOURNAL OF CHEMICAL SOCIETY
JOURNAL OF THE INSTITUTE OF METALS
JOURNAL OF MATERIALS SCIENCE
JOURNAL OF METALS
JOURNAL OF RESEARCH OF THE NATIONAL BUREAU OF STANDARDS
PART A) PHYSICS AND CHEMISTRY
PART B) MATHEMATICAL SCIENCE
JOURNAL OF THE AIR POLLUTION CONTROL ASSOCIATION
JOURNAL OF THE AMERICAN CERAMIC SOCIETY
JOURNAL OF THE AMERICAN CHEMICAL SOCIETY
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U. S. Department of Commerce
Frederick B. Dent
Secretary

National Bureau of Standards
Richard W. Roberts, Director

ANNEX IV

National Bureau of Standards Certificate of Analysis

Standard Reference Material 1631

Sulfur in Coal

Rolf A. Paulson

This Standard Reference Material is intended primarily for use as an analytical standard for the determination of sulfur in coal. It is also certified for ash content. This standard consists of three different low-volatile bituminus coals, ground to pass a 60-mesh sieve, packaged separately. Each coal is certified for its sulfur and ash contents on an as-received basis.

Coal	Source of Material	Percent by Weight	
		Sulfur	Ash
A	Keystone Mine No. 2, West Virginia	0.546 ± 0.003	5.00 ± 0.02
B	Colver Mine, Pennsylvania	2.016 ± .014	14.59 ± .09
C	Stigler Bed, Arkansas	3.020 ± .008	6.17 ± .02

The certified values are the means of 20 determinations of sulfur and 10 determinations of ash on 10 samples selected randomly from the lot of 2500 samples. The uncertainty represents the half widths of the 95% confidence intervals of the certified values. There was no evidence of heterogeneity of composition within the uncertainty limits reported.

The coals have been analyzed by five cooperating laboratories with results consistent with the certified values. All of the analytical work is summarized under the supplementary information.

The overall coordination of the technical measurements leading to certification, was under the chairmanship of J. K. Taylor.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by C. L. Stanley.

Washington, D. C. 20234
August 15, 1973
(Revised December 6, 1974)

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

Supplementary Information

Analysis of Material

The methods of analysis used for certifying this material were essentially those identified as ASTM method D271. The following laboratories cooperated with NBS in the analysis of these coals: Association Technique de l'Importation Charbonniere, Hampton Roads Laboratory, Newport News, Virginia; Combustion Engineering Inc., Windsor, Connecticut; Eastern Associated Coal Corporation, Pittsburgh, Pennsylvania; Joint Research Center, Research Establishment, CCR, Ispra Italy; and U. S. Bureau of Mines, Coal Analysis Laboratory, Pittsburgh, Pennsylvania.

Summary of Supporting Analytical Values

Coal	Laboratory	Sulfur, %	Ash, %
A	1	0.540 ± 0.006	4.847 ± 0.044
	2	.579 ± .006	4.792 ± .074
	3	.551 ± .016	5.134 ± .085
	4	.569 ± .017	4.865 ± .051
	5	.547 ± .002	
B	1	1.972 ± .016	14.50 ± .16
	2	2.019 ± .014	14.58 ± .06
	3	1.969 ± .031	14.61 ± .19
	4	1.988 ± .028	14.58 ± .16
	5	1.993 ± .008	
C	1	3.018 ± .018	6.126 ± .031
	2	3.035 ± .031	6.013 ± .092
	3	2.915 ± .017	6.092 ± .056
	4	2.998 ± .020	6.045 ± .072
	5	3.023 ± .005	

For the work performed in the cooperating laboratories the values for sulfur are the averages of twelve determinations for each laboratory except laboratory No. 5, whose values are based upon 20 determinations for Coal A and 23 determinations for coals B and C. The values for ash are the averages of twelve determinations except laboratory No. 1, whose values are based upon 18 determinations for coals A and C and 17 determinations for coal B. The uncertainties are the 95 percent confidence limits.

The Joint Research Center, Research Establishment, CCR reported mercury and lead values for coals A and B. The values were: mercury, 0.073 and 0.041 ppm and lead, 5.44 and 5.97 ppm for Coals A and B, respectively.

Originally, the moisture content of these coals was to be certified; however, the lack of homogeneity in this respect prevented certification. Therefore, these values are reported for information only. The averages including the moisture values from five of the participating laboratories were: Coal A, 0.84 percent; Coal B, 0.69 percent; and Coal C, 0.47 percent. The range of values were: Coal A, 0.62 to 1.17 percent; Coal B, 0.18 to 1.06 percent; and Coal C, 0.15 to 0.87 percent.

These coals were procured and ground through the assistance and courtesy of David E. Wolfson and Forrest E. Walker, U. S. Bureau of Mines, Pittsburgh, Pennsylvania.

Use of Material

All analytical values are reported on an as-received basis so that no drying procedures should be used. The coals are packaged in hermetically sealed envelopes each containing approximately 3g of the material. It is recommended that the envelopes be opened only at the time of analysis and that any unused contents be discarded.

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